

RUDIN, V.D., dots.; STEPANOV, P., red.; LOBKOV, M., red.

[Trace element fertilizers and crops] Mikroudobrenia i  
urozhai.

[Trace elements in animal husbandry] Mikroelementy v zhivotnovodstve. Stavropol', Stavropol'skoe knizhnoe izd-vo,  
(MIRA 18:8)  
1964.

1. Stavropol'skiy sel'skokhozyaystvennyy institut (for  
Rudin).

USSR / Soil Science. Physical and Chemical Properties  
of Soil.

J

Abs Jour: Ref Zhur-Biol., No 2, 1959, 6079.

Author : Rudin, V. D.; Shcherbakova, S. S.

Inst : Not given.

Title : The Cobalt Content of Several Soils of the  
Stavropol'skiy Kray.

Orig Pub: Materialy po izuch. Stavropol'sk. kraya, Vyp. 8,  
1956, 349-354.

Abstract: For the determination of cobalt in soil a colorimetric method was employed using nitroso-R salt (1-nitro-2 naphthol-3,6, sodium disulphonate). The latter forms with cobalt a characteristic complex compound of orange-red coloration. Fe, Ni, Cu, Mn and Zn do not interfere with the use of R-nitroso salt. Data is cited as to the co-

Card 1/2

25

USSR / Soil Science. Physical and Chemical Properties. J  
of Soil.

Abs Jour: Ref Zhur-Biol., No 2, 1959, 6079.

Abstract: Cobalt content of various Stavropol' soils. The greatest cobalt content was observed in Cis-Caucasian chernozem soils with an average thickness (0.90-0.31), while the smallest amount was observed in brown sandy soils (0.35 - 0.05 mg. per 100 grams of soil). In samples of investigated soils the cobalt content in the tillable horizon A was everywhere greater than in horizon B. Sinking deeper into horizon C, the cobalt content increases. This explains the loss of cobalt by the plant roots. Bibliography contains 8 titles.  
-- L. N. Pankratova.

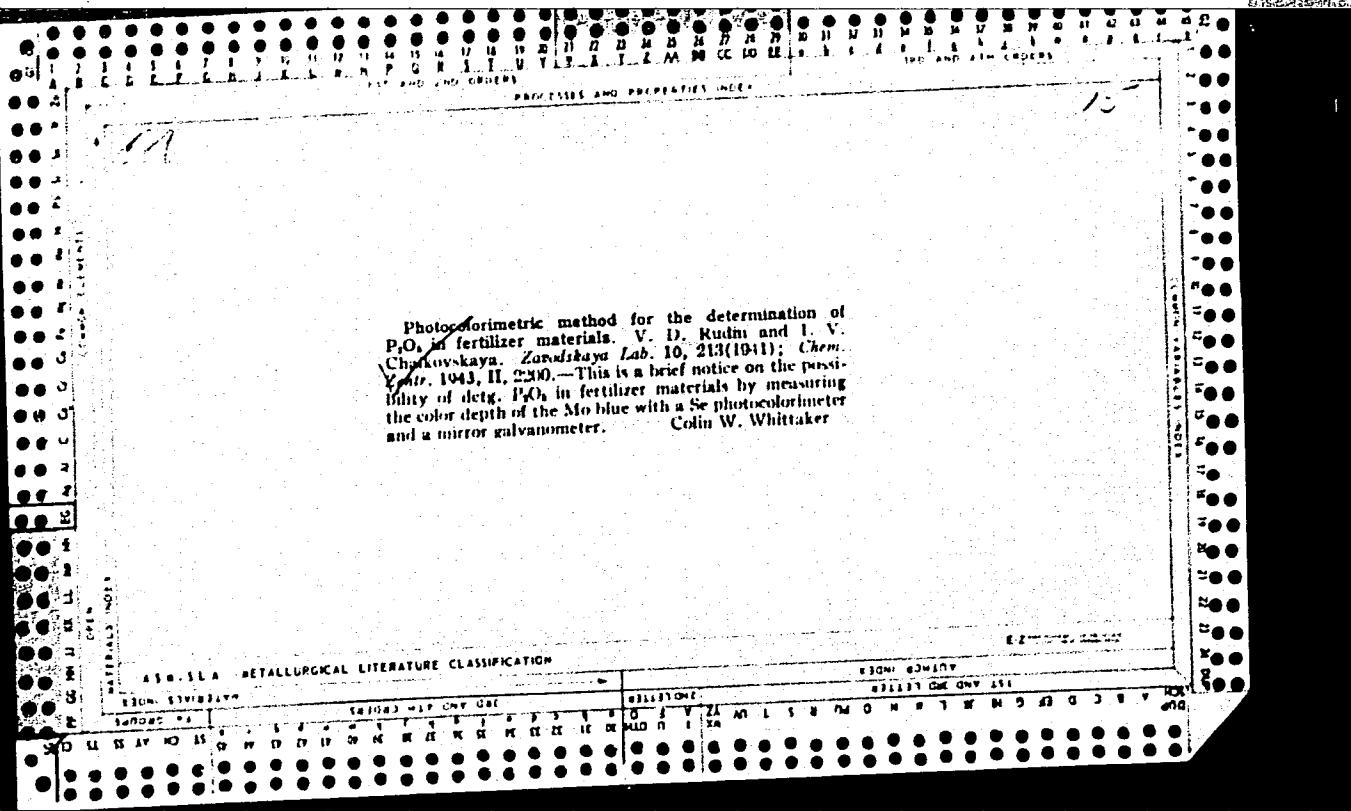
Card 2/2

RUDIN, V.D.

Effect of trace elements on grain yields and quality in different farm crops. Nauch. dokl. vys. shkoly; biol. nauki no.3:143-146  
'61. (MIRA 14:7)

1. Rekomendovana kafedrami farmakologii i biologicheskoy, organicheskoy i fizicheskoy kolloidnoy khimii Stavropol'skogo sel'skokhozyaystvennogo instituta.

(STAVROPOL TERRITORY—GRAIN)  
(TRACE ELEMENTS)



ACC NR: AP6033445

SOURCE CODE: UR/0051/66/021/004/0522/0523

AUTHOR: Rudin, V. L.

ORG: none

TITLE: Use of an optical-acoustic method for the measurement of absorption of electromagnetic energy in gases at centimeter wavelengths

SOURCE: Optika i spektroskopiya, v. 21, no. 4, 1966, 522-523

TOPIC TAGS: optic method, acoustic effect, electromagnetic wave absorption, gas absorption

ABSTRACT: The author describes an experiment which demonstrates the feasibility of measuring the absorption of electromagnetic energy in gases at microwave frequencies by using an optical-acoustic method wherein the acoustic oscillations in a gas, corresponding directly to the energy absorbed in the gas, are observed directly. The experiments make it possible to compare the change in the signal at the output of a crystal detector with the signal developed by the optical-acoustic receiver at different concentrations of the absorbing gas in air, and to demonstrate that the optical-acoustic method has advantages in the sense of producing a larger change in signal in response to a change in concentration. The gas tested was Freon-22 ( $\text{CHClF}_2$ ). The absorption was produced in a resonator which served as part of the optical-acoustic equipment (Fig. 1), and is a modification of apparatus described by the author earlier (Opt. i spektr. v. 20, 364, 1966). The results showed that the signal developed by

Card 1/2

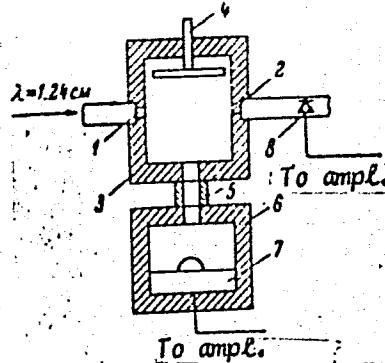
UDC: 535.34 - 1

ACC NR: AP6033445

Fig. 1. Measurement setup. 1,2 - Resonant diaphragms, 3 - electromagnetic cavity resonator, 4 - tuning plunger, 5 - tube, 6 - acoustic resonator, 7 - electrodynamic microphone, 8 - crystal detector

the optical-acoustic receiver was much larger than the relative change in signal obtained with a crystal detector. The maximum signal is obtained at an approximate concentration 50 - 70%. Allowance for the change in the specific heat of the gas during the experiment shifts the maximum to the region of 30% concentration but does not change the character of the dependence of the absorption on the concentration. Other advantages and possible applications of the method are discussed. The author thanks M. L. Veyngarov for directing the work. Orig. art. has: 2 figures and 1 table.

SUB CODE: 20/ SUBM DATE: 26Apr66/ ORIG REF: 004/ OTH REF: 002



Card 2/2

L 20178-66 EMT(d)  
ACC NR: AP6007021

SOURCE CODE: UR/0051/66/020/002/0364/0365

AUTHOR: Rudin, V. L.

20  
19  
B

ORG: none

TITLE: An opticoacoustic receiver for centimeter waves *(445)*

SOURCE: Optika i spektroskopiya, v. 20, no. 2, 1966, 364-365

TOPIC TAGS: opticoacoustic receiver, centimeter wave, acoustic resonance

ABSTRACT: The author describes a highly sensitive opticoacoustic receiver designed for quantitative studies in the centimeter wavelength range. The device consists of a centimeter wave generator ( $\lambda = 1.25$  cm), a waveguide channel, an opticoacoustic receiver, an amplifier, a synchronous detector, and d-c recording device. The receiver contains a system which consists of a resonator for centimeter waves and a resonator for the acoustic oscillations generated by pressure pulsations in the gas (see figure). The centimeter waves pass through a resonance iris which has a mica window 1 into the resonator 2 tuned to their frequency. Piston 3 is used for tuning.

UDC: 621.317.794-14

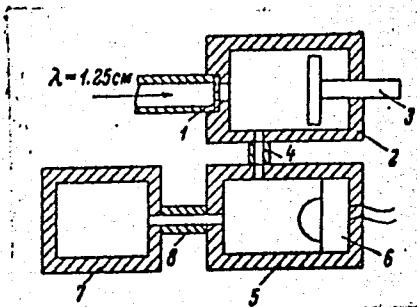
2

Card 1/2

L 20178-66

ACC NR: AP6007021

The resonator is coupled by connection tube 4 to vessel 5 containing moving-coil microphone 6. This system is a double resonator for acoustic oscillations. The frequency response of the microphone is used for calculating the diameter and length of tube 4, which determine the resonance properties of the system. The resonance properties are improved by using an additional resonator 7 and tube 8 which amplify the useful signal. The signal-to-noise ratio for the instrument was greater than 100 when a mixture of freon and air was used (3% freon) and the time constant for the measuring device was 0.25 sec. "The author thanks M. L. Veyngerov for directing this work." Orig. art. has: 2 figures.



SUB CODE: 20/ SUBM DATE: 18Jun65/ ORIG REF: 004/ OTH REF: 002  
ATD PRESS: 4214

Card 2/2 *MJS*

[14]

Country : USSR

Title : Soil Science, Physical and Chemical Properties  
of Soil

Author : V. A. Krasil'nikov et al., 1979

Editor : Radjib, V.L.; Shcherbakova, S/S/

Inst. : Stavropol' Agric. Inst.

Subject : Manganese and Molybdenum Microelement Content  
in the Soils of Stavropol'skiy Kray and Their  
Effect on the Corn Yield Boost.

Publ. Place : Tr. Stavropol'skiy agro. in-ta, 1955, vyp. 7,  
53-62

Abstract : The Mn content in soils of the Stavropol'skiy  
Kray ranges within the limits of  $2.01 \cdot 10^{-2}$  to  
 $7.86 \cdot 10^{-2}$ %. Chernozem, dark-chestnut and  
Solonetz soils have more Mn than the soils on  
the shores of the Caspian Sea. The soil upper  
horizons contain more Mn than the lower ones.  
The largest Mo content was found in Solonetz  
soils,  $10.8 \cdot 10^{-4}$ %. In other soils its content  
varies between  $7.10 \cdot 10^{-4}$  to  $7.0 \cdot 10^{-4}$ %. The  
smallest Mo content was found in dark

CARD : 1/2

Technical Report

Report, Jurnal Sistem Penerapan, No. 5, 1951, Nov., 20079

Abstract: The results of experiments on the use of micro-elements in agriculture in the U.S.S.R. are presented. It is shown that the use of micro-elements in the soil increases the yield of corn by 6.5 to 9.7%, according to S.A. Nikitin. The author also presents the results of experiments on the use of micro-elements in the soil for the cultivation of corn, which have an average yield increase of 6.5 to 9.7%. The placement of these micro-elements in the soil increased the yield of corn by 6.5 to 9.7%, according to S.A. Nikitin.

"APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0

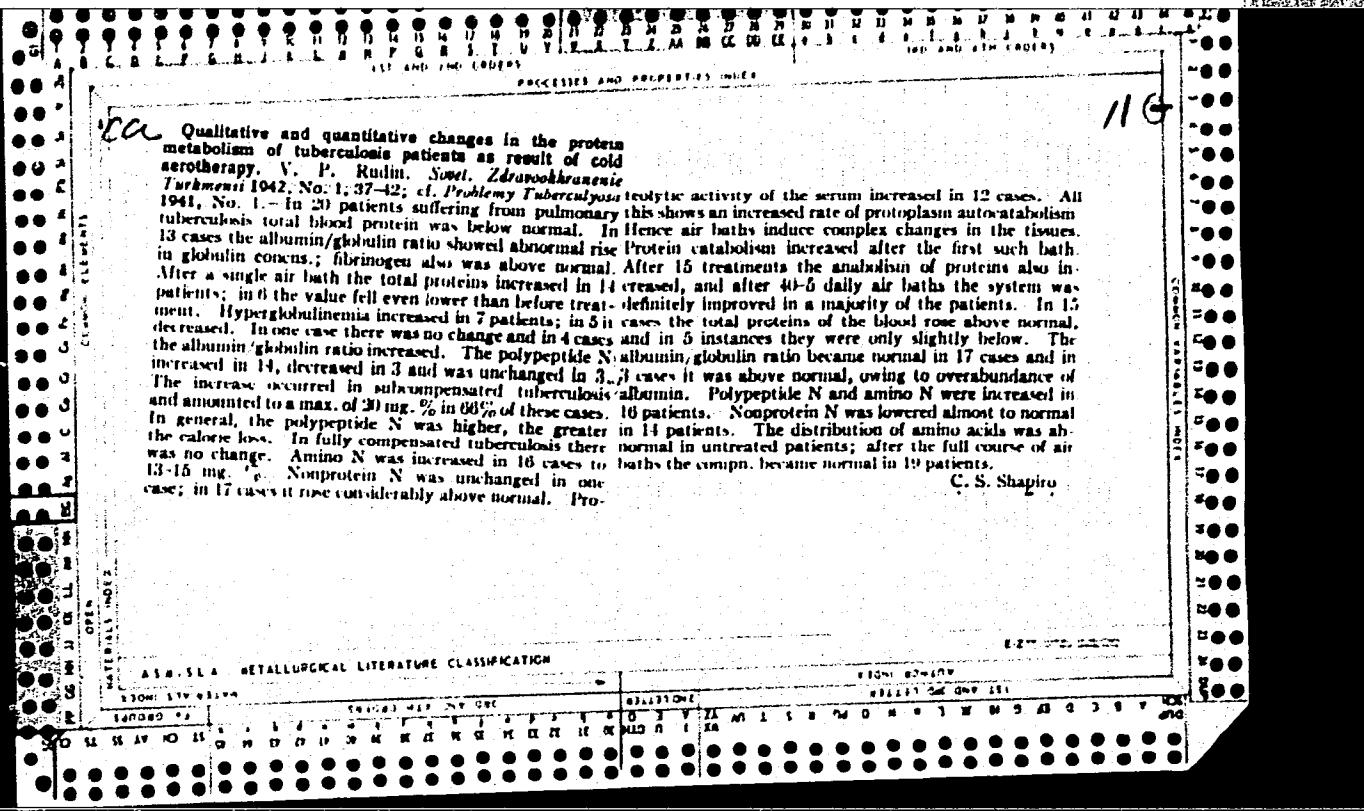
GOREGINSKY, G.M., kand. tekhn. nauk, RUDNIN, V.I., inst.

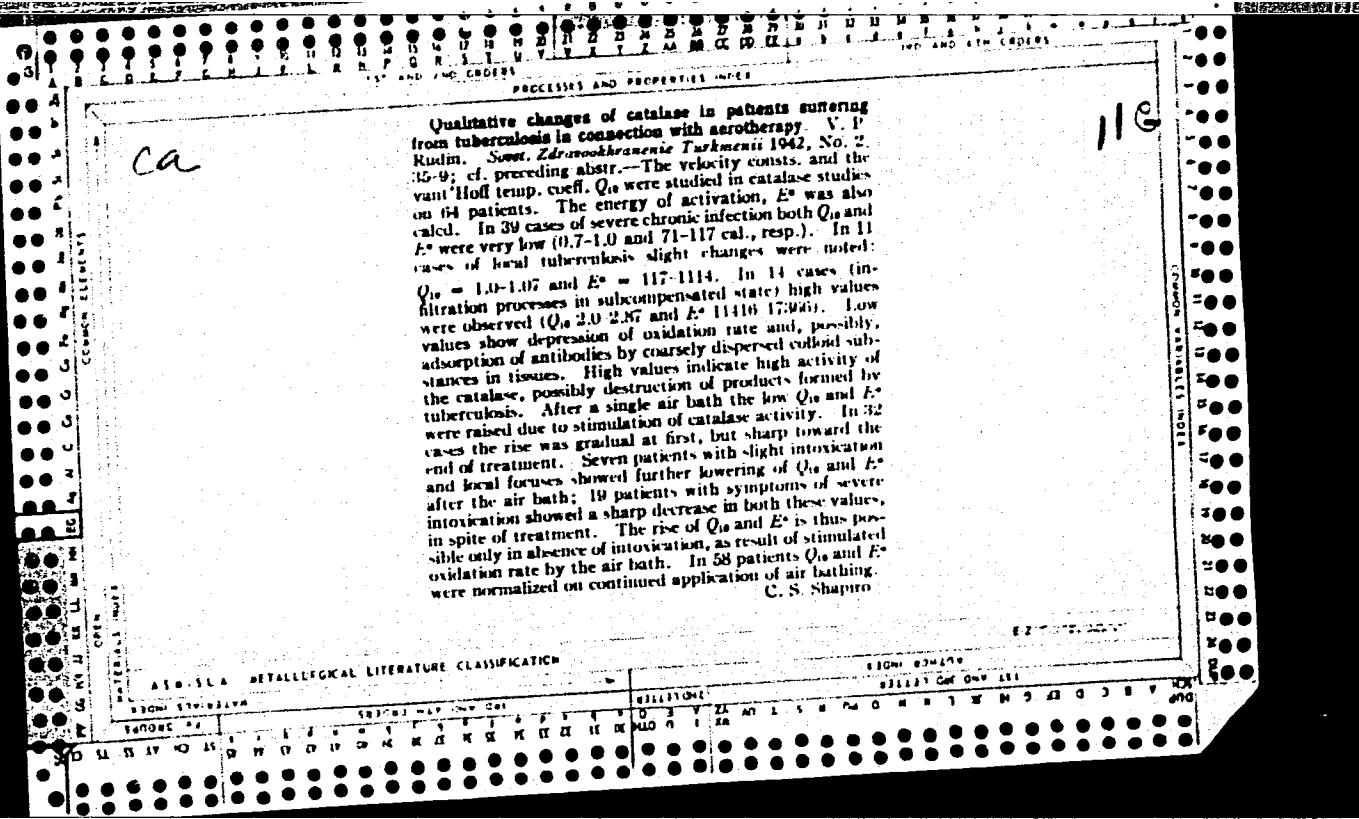
Laboratory control method of the roughness of ground glass surfaces. Stek. i ker. 22 no.4:28-29 Ap '65. (MIRA 18:5)

I. Leningradskiy institut tekhniki mekhaniki i optiki.

APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0"





Qualitative changes, in pulmonary tuberculosis, of catalase and their displacement in treatment with aerobaths in the cold season. V. P. Rudin. *Russ. Akad. Med. Nauk. Med. Moshchn. No. 9, 71-4 (1912); cf. C. I. 38, 1024.*  
The van't Hoff temp. coeff., and activation energy of catalase show variations in pulmonary tuberculosis, in proportion to the degree of intoxication and reactivity of the organism. The values of these factors can be used as rather sensitive indexes for the estn. of organism reactivity in the course of cold aerotherapy. In a majority of cases cold therapy activates the oxidation-reduction system and raises the stability of the organism to the tuberculous toxin.  
G. M. Koslapoff

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ASB-SEA METALLURGICAL LITERATURE CLASSIFICATION

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081831 Oct 047 196

APPROVED FOR RELEASE: 06/20/2000

**CIA-RDP86-00513R001445920015-0"**

RUBIN, V. P.

Investigation of heterogeneous equilibria of lithium chloride with the chlorides of the metals of the second group.  
V. P. Rubin: *Izvest. Akad. Nauk S.S.R., Otdel. Khim. Nauk*, 1954, 400-9; cf. *C.A.* 48, 13393a. — The isotherms of the systems LiCl-MCl-H<sub>2</sub>O were detd. at 25° and 40° for M = Li, Ca, Sr, Ba, Zn, and Cd; for M = Hg the isotherm at 30° was obtained. The systems with M = Be, Ca, Sr, Ba, and Hg did not form any compds. The systems with Zn and Cd chlorides formed ZnCl<sub>4</sub>·2LiCl·5H<sub>2</sub>O and LiCl·CuCl<sub>2</sub>·2.5H<sub>2</sub>O. The presence of LiCl increased the solv. of HgCl.  
I. Bencowitz

RUDIN, V.P., professor; DOBROVINSKAYA, Ye.K.; PODDUBN'Y, A.F.

Errors in diagnosing tuberculous meningitis in adults. Vrach. delo  
no.3:227-231 Mr '57 (MLRA 10:5)

1. Kafedra fiziologii (zav.-prof. V.P. Rudin) Kiyevskogo  
meditsinskogo instituta.  
(MENINGITIS--TUBERCULOSIS)

RUDIN, V.P., professor; OKHRIMENKO, R.D.

Acute miliary tuberculosis in young people. Vrach.delo no.7:701-705  
Jl '57. (MLRA 10:8)

1. Kafedra ftiziatrii (zav. - prof. V.P.Rudin) Kiyevskogo meditsinskogo instituta  
(TUBERCULOSIS)

OKREMA, V.Ya.; RUDIN, V.P., prof., nauchnyy rukovoditel' raboty.

Immediate and late results of antibacterial treatment of pulmonary tuberculosis in elderly and senile patients. Sov. med. 27 no.2:14-17 F '64. (MIRA 17:10)

1. Tul'chinskiy protivotuberkuleznyy dispanser (glavnnyy vrach V.Ya. Okrema) Vinnitskoy oblasti.

RUDIN, V.P., prof. (Kiyev)

Differential diagnosis of lung cancer and tuberculosis. Vrach.  
delo no.9:85-89 S '61. (MIRA 14:12)

1. Ukrainskiy nauchno-issledovatel'skiy institut tuberkuleza.  
(LUNGS—CANCER) (TUBERCULOSIS—DIAGNOSIS)

RUDIN, V.P., prof.; OKHRIMENKO, R.D.

Acute miliary tuberculosis in young persons. Pat., klin.i terap.  
tub. no.8:192-196 '58. (MIRA 13:7)

1. Iz kafedry ftiziatrii (zav. - prof. V.P. Rudin) Kiyevskogo  
meditsinskogo instituta im. akad. A.A. Bogomol'tsa.  
(TUBERCULOSIS)

RUDIN, V.P., prof.

Early detection and treatment of tuberculosis in diabetes mellitus patients. Vrach.delo no.11:1163-1167 N '59. (MIRA 13:4)

1. Kafedra ftiziatrii (zaveduyushchiy - prof. V.P. Rudin) Kiievskogo meditsinskogo instituta.  
(TUBERCULOSIS) (DIABETES)

RUDIN, V.P., prof.

Pulmonary tuberculomas. Vrach.delo no.8:799-805 Ag '58 (MIRA 11:8)

1. Kafedra ftiziatrii (zav. - prof. V.P. Rudin) Kiyevskogo meditsinskogo  
instituta.

(LUNGS--TUMORS)

RUDIN, V.P., prof.; MARTYNOVA, S.I., starshaya meditsinskaya sestra

Organizing care of patients with tuberculous meningitis. Med.  
sestra 16 no.11:18-21 N '57. (MIRA 11:2)

1. Kafedra ftiziatrii Kiyevskogo meditsinskogo instituta.  
(MENINGES--TUBERCULOSIS)

RUDIN, V.P., professor; SIN'KOVSKIY, P.V.; PODDUBNYY, A.F.

Cardiovascular system in tuberculous meningitis in adults.  
Pat., klin.i terap.tub. no.8:58-63 '58. (MIRA 13:7)

1. Iz kafedry tuberkuleza (zav. -- prof. V.P. Rudin) Kiyevskogo  
meditsinskogo instituta im. akad. A.A. Bogomol'tsa.  
(MENINGES--TUBERCULOSIS) (CARDIOVASCULAR SYSTEM)

RUDIN, V.P., professor

Mixed form of tuberculous meningitis in adults. Pat., klin. i terap.tub. no.8:64-67 '58. (MIRA 13:7)

1. Iz kafedry ftiziatrii (zav. - prof. V.P. Rudin) Kiyevskogo meditsinskogo instituta im. akad. A.A. Bogomol'tsa.  
(MENINGES--TUBERCULOSIS)

RUDIN, V.P., prof.

Digestive apparatus in tuberculous meningitis in adults. Pat.,  
klin.i terap.tub. no.8:68-74 '58. (MIRA 13:7)

1. Iz kafedry ftiziatrii (zav. - prof. V.P. Rudin) Kiyevskogo  
meditsinskogo instituta im. akad. A.A. Bogomol'tsa.  
(MENINGES--TUBERCULOSIS) (DIGESTIVE ORGANS)

RUDIN, V.P., professor; GRIB, N.I.

Blood transfusion as a method of stimulating therapy for patients with tuberculosis. Pat., klin.i terap.tub. no.8:162-165 '58.

(MIRA 13:7)

1. Iz kafedry ftiziatrii (zav. - prof. V.P. Rudin) Kiyevskogo meditsinskogo instituta im. akad. A.A. Bogomol'tsa.  
(BLOOD--TRANSFUSION) (TUBERCULOSIS)

HUDIN, V.P., prof.; TROFIMETS, V.V., assistant

Clinical aspects and treatment of spontaneous pneumothorax.  
Pat., klin.i terap.tub. no.8:179-182 '58. (MIRA 13:7)

1. Iz kafedry ftiziatrii (zav. - prof. V.P. Rudin) Kiyevskogo  
meditsinskogo instituta im. akad. A.A. Bogomol'tsa.  
(PNEUMOTHORAX) (TUBERCULOSIS)

HUDIN, V.P., professor; OKHRIMENKO, R.D.

Miliary tuberculosis in middle-aged and old people. Pat., klin.  
i terap.tub. no.8:187-191 '58. (MIRA 13:7)

1. Iz kafedry ftiziatrii (zav. - prof. V.P. Rudin) Kiyevskogo  
meditsinskogo instituta im. akad. A.A. Bogomol'tsa.  
(TUBERCULOSIS)

RUDIN, V.Ya.; YARYM-AGAYEV, N.L.

Method for calculating the crystallization region of sodium chloride  
in the quaternary system  $\text{CaCl}_2-\text{MgCl}_2-\text{NaCl}-\text{H}_2\text{O}$ . Zhur.prikl.khim.  
30 no.6:941-944 Je '57. (MIRA 10:10)

1. Donetskiy industrial'nyy institut imeni N.S.Khrushcheva.  
(Sodium chloride) (Crystallization) (Systems (Chemistry))

YARYM-AGAYEV, N.L.; RUDIN, V.Ya.; TSEYTLENOK, T.A.

Refractometric determination of the composition of solutions containing potassium chloride and sodium nitrate. Zhur.anal.khim. no. 6:701-705 Je '63. (MIRA 16:9)

1. Donetsk Polytechnical Institute.  
(Potassium chloride) (Sodium nitrate) (Refractometry)

Rudin, V. Ya.

USSR/Physical Chemistry - Thermodynamics, Thermochemistry, Equilibria,  
Physical-Chemical Analysis, Phase Transitions.

B-8

Abs Jour: Referat. Zhurnal Khimiya, No 3, 1958, 7171.

Author : V. Ya. Rudin, N.L. Yarym-Agayev.

Inst :

Title : Computation Method of Sodium Chloride Crystallization Zone  
in Quaternary System  $\text{CaCl}_2 - \text{MgCl}_2 - \text{NaCl} - \text{H}_2\text{O}$ .

Orig Pub: Zh. prikl. khimii, 1957, 30, No 6, 941-944.

Abstract: A computation method for the determination of salt concentrations of the quaternary system  $\text{CaCl}_2 - \text{MgCl}_2 - \text{NaCl} - \text{H}_2\text{O}$  in the crystallization zone of NaCl is proposed. Known values of NaCl solubility in ternary systems  $\text{CaCl}_2 - \text{NaCl} - \text{H}_2\text{O}$  and  $\text{MgCl}_2 - \text{NaCl} - \text{H}_2\text{O}$  are used in this method. The computed values differ from experimental ones by magnitudes which do not exceed experimental errors.

Card : 1/1

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CIA-RDP86-00513R001445920015-0

YAKYMAGAYEV, N.L.; RUDIN, V.Y.; TSEYTLENOK, T.A.

Salt solubility isoler in the system K, Na //Cl<sub>2</sub> No<sub>3</sub> - H<sub>2</sub>O.  
Zhur.neorg.khim. 10 no.4:976-980 Ap '65. (MIRA 18:6)

APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0"

YARYM-AGAYEV, N.L.; KOGAN, Ye.A.; RUDIN, V.Ya.; TITOVA, V.A.

Orthobaric heats of mixing of pyridine and acetic acid vapors. Zhur.fiz.  
khim. 37 no.7:1445-1449 J1 '63. (MIRA 17:2)

1. Donetskiy politekhnicheskiy institut.

YARYM-AGAYEV, N.L.; RUDIN, V.Ya.; TITOVA, V.A.; KOGAN, Ye.A. (Stalino)

Orthobaric heats of mixing of pyridine and water vapors. Zhur.-fiz.khim. 35 no.10:2285-2290 O '61. (MIRA 14:11)

1. Donetskij politekhnicheskiy institut.  
(Pyridine) (Water vapor) (Heat of mixing)

5(4)

AUTHORS:

Rudin, V. Ya., Yarym-Agayev, N. L.

SOV/78-4-3-26/34

TITLE:

On the Form of the Isothermal Lines in the Case of a Variation  
of the Molar Properties of the Binary System With Formation  
of Rational Compounds (O forme izoterm izmeneniya  
mol'nego svoystva dvoynykh sistem s obrazovaniyem ratsional'-  
nogo soyedineniya)

PERIODICAL:

Zhurnal neorganicheskoy khimii, 1959, Vol 4, Nr 3,  
pp 662-670 (USSR)

ABSTRACT:

The variation of the isothermal lines of the molar properties  
of the binary system in which the rational compound S  
forms from the components A-B was investigated. The general  
equation (19):

$$G_{A-B} = G_A^* \frac{s}{n} N_B + G_{A-S} \frac{n - (m + n - s)N_B}{n}$$

gives the dependence between the molar composition and the  
molar properties in the system A-B in the case of formation  
of the rational compound S. It was shown that the  
branch isothermal lines A-B and A-S are adiabatic. The iso-  
thermal line A-B corresponds with or without maximum to the

Card 1/2

On the Form of the Isothermal Lines in the Case  
of a Variation of the Molar Properties of the Binary System With Formation of  
Rational Compounds

SOV/78-4-3-26/34

Isothermal line A-S with a maximum. In the case of formation of the compound S a maximum or a minimum may occur. Isothermal lines that do not show a bending point in the case of a variation of the molar composition have a maximum on the branch isothermal lines A-B. The behavior of several types of branch isothermal lines was discussed in the case of variation of the molar composition in the system A-B and is given in table 1. There are 3 figures, 1 table, and 3 Soviet references.

ASSOCIATION: Donetskiy industrial'nyy institut im. N. S. Khrushcheva  
(Donetskiy Industrial Institute imeni N. S. Khrushchev)

SUBMITTED: April 17, 1957

Card 2/2

"APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0

YARYM-AGAYEV, N.L.; KLYASHTORNAYA, F.M.; RUDIN, V. Ya.

Aqueous system of potassium and sodium nitrates and chlorides.  
Zhur. neorg. khim. 9 no.11:2639-2644 N '64 (MIRA 18:1)

APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0"

AID P - 3752

Subject : USSR/Chemistry  
Card 1/1 Pub. 152 - 16/22  
Authors : Uspenskaya, L. N., A. G. Bergman, and V. Ya. Rudin  
Title : Effect of thermal conditions under which the manufacture of "red ferric oxide" from ferrous sulfate takes place on the change of the hue of its color  
Periodical : Zhur. prikl. khim. 28, 1006-1009, 1955  
Abstract : Removal of water and decomposition of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  were studied at 600, 650, 700, 750 and  $800^\circ\text{C}$  over periods of from 2-10 hours. Samples containing less than 95%  $\text{Fe}_2\text{O}_3$  are darker than those containing over 96%  $\text{Fe}_2\text{O}_3$ . Higher temperatures ( $750-800^\circ\text{C}$ ) and prolonged heating (9-10 h:s.) cause darkening of the samples due to increase in the size of the  $\text{Fe}_2\text{O}_3$ -particles. Two diagrams, 3 references, all Russian (1944-1949).  
Institution : Rostov-na-Donu State University im. V. M. Molotov  
Submitted : Ja 3, 1954

RUDIN, V.Ya.; YARYM-AGAYEV, N.L.

Shape of isotherms of change in the molar property of binary systems which form proportional compounds. Zhur. neorg. khim. 4 no.3:662-670 Mr '59. (MIRA 12:5)

1. Donetskij industrial'nyy institut im. N.S. Krushcheva.  
(Systems (Chemistry))

"APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0

APPROVED FOR RELEASE: 06/20/2000 CIA-RDP86-00513R001445920015-0"

RUBIN, Ia.II., bugermeyster-pastavnik; MLYUNIN, V.G.

Working rock with a multiarket suction dredge. Transp. siroi.  
15 no.1:20-23 ja '65. (MIRA 16:3)

i. Nachal'nik temlecherpatel'nogo karavana "Volkhov" (for  
Mlyunin).

LAVRINOVICH, Ye.V., kand. tekhn. nauk; GOLITSYNSKIY, D.M., inzh.;  
PERLEV, Ye.M., inzh.; RUDIN, Ye.I., inzh.

Contracting vertical seams of cylindrical shells. Transp.  
stroj. 14 no.3xL5-48 Mr '64. (MIRA 17:6)

"APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0

RUDIN, Yu.S., INZIN.

Manufacture of welded housings for electric motors in place of  
cast housings. Svar.prom. no. 1223-44. 3/64.

(MIRE 1961)

APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0"

RUDIN, IU. V.

Iz praktiki remonta porshnevых паровых машин. (Moskva) Gizlegprom, 1946.  
26 p. illus. (Bibliotekha tekstil'shchika)

Practice in repairing reciprocating steam engines.

DLC: TJ471. R8

SO: Manufacturing and Mechanical Engineering in the Soviet Union, Library  
of Congress, 1953.

DEMISOV, Nikolay Mitrofanovich; SAMOYLENKO, P., otv. red.; VARNAKOVA, N.,  
red.; RUDINA, G., red.; YURMANOVA, A., red.

[Reinforced-concrete supports and their use in Kuznets Basin  
mines] Zhelezobetonnaia krep' i ee primenenie na shakhtakh  
Kuzbassa. Kemerovo, Kemerovskoe knizhnoe izd-vo, 1959. 177 p.  
(MIRA 15:11)

(Kuznetsk Basin--Mine timbering)  
(Reinforced concrete construction)

RUDINA, L. A.: Master Biol Sci (disc) -- "The nutrition conditions and structure of the sod-meadow soils of the flood plain of the Moscow River". Moscow, 1959. 20 pp (Moscow Order of Lenin and Order of Labor Red Banner State Univ M. V. Lomonosov, Soil-Biol Faculty), 120 copies (KL, No 17, 1959, 107)

RUDINA, L.A.

Dynamics of mobile nitrogen, phosphorus, and potassium compounds in  
turf-meadow soils of the Moskva River bottom lands. Vest.Mosk.un.  
Ser.biol., pochv., geol., geog. 13 no.3:91-100 '58.

(MIRA 12:1)

1. Kafedra pochvovedeniya Moskovskogo gos. universitata.  
(Moskva Valley--Soil chemistry)

RUDINA, L.A.

Dynamics of water-stable structure in the top layer of Turf-Meadow soils in bottom lands of the Moskva River. Nauch.dokl. vys.shkoly;biol.nauki no.4:179-183 '58. (MIRA 11:12)

1. Rekomendovana kafedroy pochvovedeniya Moskovskogo gosudarstvennogo universiteta imeni M.V.Lomonosova.  
(Moskva Valley--Soil structure)

YEVDOKIMOVA, T.I.; RUDINA, L.A.

Role of the herbaceous vegetation in the process of soil formation  
in the Moskva River floodland [with summary in English].  
Pochvovedenie no. 9:80-88 '58. (MIRA 11:10)

1. Moskovskiy gosudarstvennyy universitet imeni M.V.Lomonosova.  
(Moskva Valley--Soil formation)

RUDINH L.F.

f-2

PHASE I BOOK EXPLOITATION

846

U.S.S.R. Ministerstvo geologii i okhrany nedr

Metody opredeleniya radioaktivnykh elementov v mineral'nom syr'ye  
(Methods of Determining Radioactive Elements in Mineral Raw  
Materials) Moscow, Gosgeoltekhnizdat, 1958. 68 p. 3,000 copies  
printed.

Compilers: Sochevanov, V.G. and Titov, V.I.; Ed.: Krasnova, N.E.  
Tech. Ed.: Averkiyeva, T.A.

PURPOSE: This book is for those engaged in geochemical prospecting  
for radioactive ores.

COVERAGE: The chemical determination of radioactive substances in minerals and rock formations is described in this publication. Chemical treatment of materials in preparation for radiometric analysis is also included. The proposed methods are considered to be the most

Card 1/4

## Methods of Determining Radioactive Elements (Cont.) 846

reliable for geochemical research. Methods are presented in the form of separate procedure instructions with the inclusion of: principle of the method, elimination of interfering factors, application limits, necessary reagents, procedure of analysis. Specifications for high purity reagents are given whenever necessary. There is a bibliography with 26 references, 17 of which are Soviet, 4 English, 3 German, 1 Czech, and 1 Swiss.

## TABLE OF CONTENTS:

Preface

3

## URANIUM

Titov, V.I., Volkov, I.I. Colorimetric Determination of Uranium  
by the Trilon-Phosphate Method

4

Zvenigorodskaya, V.M., Rudina, L.P. Colorimetric Determination of  
Uranium by Means of the Fluoride Method

12

Card 2/4

"APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0

RUDINA, M.A., arkhitektor

Rolled materials made of polymers for interior finishing of walls.

Sbor. trud. VNIINSM no.7:151-158 '63.

(MIRA 17:11)

APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0"

LOSKUTOVA, L.T.; MAKOTINSKIY, M.P., kand. arkh.; RUDINA, M.A., arkh.; SHPANOV, I.A., arkh. Prinimal uchastiye LIVSHITS, A.M., inzh.; GROMOV, V.L., kand. tekhn. nauk, retsenzeng; KRASNOVSKIY, N.V., kand. tekhn. nauk, retsenzent; PAVLOV, V.P., kand. tekhn. nauk, retsenzent; PODZOROVA, N.G., inzh., retsenzent; FOLOMIN, A.I., doktor tekhn. nauk, retsenzent; CURVICH, E.A., red.

[Catalog of finishing materials and elements] Katalog otdelochnykh materialov i izdelii. Moskva, Gosstroizdat. Pt. 8 [Wood and paper] Derevo i bumaga. 1962. 56 p. (MIRA 16:8)

1. Vsesoyuznyy nauchno-issledovatel'skiy institut novykh stroytel'nykh materialov.

(Finishes and finishing)

KOSHKIN, V.G., kand. tekhn.nauk; MAKOTINSKIY, M.P., kand. arkh.; MUNTS, V.O., kand. arkh.; RUDINA, M.A., arkh.; SILUANOVA, G.V., arkh.; SHORYGINA, N.V., kand. khim. nauk; Primalni uchastiye: BOGUSLAVSKIY, A.I., inzh.; ZARUBITSKIY, A.Ye., inzh.; LIVSHITS, A.M., inzh.; MASHINA, N.N., inzh.; OTLIVANCHIK, A.N., kand. tekhn. nauk; ROMANOVA, L.A., inzh.; CHERKINSKIY, Yu.S., inzh.; ANDREYEV, V.S., retsenzent; IOFAN, B.M., retsenzent; KRIPPA, A.I., arkh., retsenzent; GURVICH, E.A., red.izd-va; BRUSINA, L.N., tekhn. red.

[Catalog of finishing materials and products] Katalog otdelochnykh materialov i izdelii. Moskva, Gosstroizdat. Pt.1.[Plastics; polymer finishing materials] Plastmassy; polimernye otdelochnye materialy. 1962. 119 p. (MIRA 16:4)

1. Vsesoyuznyy nauchno-issledovatel'skiy institut novykh stroitel'nykh materialov. 2. Chleny-korrespondenty Akademii stroitel'stva i arkitektury SSSR (for Andreyev, Iofan, Krippa).  
(Plastics) (Building--Details)

KOSHKIN, V.G., kand. tekhn. nauk; MAKOTINSKIY, M.P., kand. arkh.; MUNTS, V.O., kand. arkh.; RUDINA, M.A., arkh.; SILUANOVA, G.V., arkh.; SHORYGINA, N.V., kand. khim. nauk. Prinimali uchastiye: BOGUSLAVSKIY, A.I., inzh.; ZARUBITSKIY, A.Ye., inzh.; LIVSHITS, A.M., inzh.; MASHINA, N.N., inzh.; OTLIVANCHIK, A.N., kand. tekhn. nauk; ROMANOVA, L.A., inzh.; CHERKINSKIY, Yu.S., inzh.; ANDREYEV, V.S., retsenzent; IOFAN, B.M., retsenzent; KRIPPA, A.I., arkh., retsenzent; GURVICH, E.A., red.izd-va; BRUSINA, L.N., tekhn. red.

[Catalog of finishing materials and articles] Katalog ot-delochnykh materialov i izdelii. Pod red. M.P.Makotinskogo. Moskva, Gosstroizdat. Pt.1.[Plastics; polymer finishing materials and articles] Plastmassy; polimernye otdelochnye materialy i izdeliia. 1962. 119 p. (MIRA 16:4)

1. Moscow. Vsesoyuznyy nauchno-issledovatel'skiy institut novykh stroitel'nykh materialov. 2. Chlen-korrespondent Akademii stroitel'stva i arkhitektury SSSR (for Andreyev, Iofan, Krippa).

(Finishes and finishing--Catalogs) (Plastics)

9.9100

S/169/61/000/003/015/022  
A005/A005

Translation from: Referatiivnyy zhurnal, Geofizika, 1961, No. 3, p. 28, # 3G247

AUTHORS: Rudina, M. P., Serebrennikova, N. I.

TITLE: The Structure of the Ionosphere According to the Sped up Recording of Ionograms

PERIODICAL: "Tr. Sibirs. fiz.-tekhn. in-ta pri Tomskom un-te", 1959, No. 37,  
pp. 363-368

TEXT: Results are presented from a comparison of ionograms taken at Tomsk by the manual station at hourly times and ionograms recorded with intervals of 10 min by the panoramic station of the N. D. Bulatov system at stable state (March 4-5, 1956) and disturbed state (April 23) of the ionosphere. It is shown that the sped up observations by the panoramic station give a more detailed information on the ionosphere structure and high-speed processes. /B

N. P.

Translator's note: This is the full translation of the original Russian abstract.

Card i/1

86775

S/112/60/000/020/003/004  
A005/A001

9,9110

Translation from: Referativnyy zhurnal, Elektrotehnika, 1960, No. 20, pp. 12-13, # 6.16403

AUTHOR: Rudina, M.P.

TITLE: Preliminary Results From Measuring the Radiowave Absorption in the Ionosphere

PERIODICAL: Tr. Sibirs. fiz.-tekhn. in-ta pri Tomskom un-te, 1959, No. 37, pp. 394-396

TEXT: The measurements have been carried out since July 1957 at Tomsk correspondingly to the program of the International Geophysical Year. The measuring unit consists of: the transmitter covering the frequency band of 1-16 Mc with the pulse power of 1 kw, the receiver of the superheterodyne type with the 40 kc-pass-band, and the photo-recorder. The photorecording was carried out at the frequency of  $2.2 \pm 0.2$  Mc. The monthly average value of the reflection coefficient was calculated from 2 reflections at visual measurements and photorecordings in night time, on the basis of which the absorption was calculated. The coefficient of reflection from the F2-layer changed on the average in July - March, in the

Card 1/2

86775

S/112/60/000/020/003/004  
A005/A001

X  
V

Preliminary Results From Measuring the Radiowave Absorption in the Ionosphere

lightless diurnal time, in the limits from 0,4 to 1. It is shown that the value of the averaged absorption coefficient at the local noon at Tomsk was < 20 db. It is noted that a considerable increase of absorption was detected in single days in November - December and January. Hereat, the minimum reflection frequency attained in some cases 3 Mc.

V.I.B.

Translator's note: This is the full translation of the original Russian abstract.

Card 2/2

L 16852-63  
Po-4/Pe-4/Pq-4 RB/PT-2  
ACCESSION NR: AR3006327

EWT(1)/BDS/EEC-2/ES(v) AFFTC/ASD/AFMDC/ESD-3/APGC Pi-4/  
S/0058/63/000/007/H030/H030

SOURCE: RZh. Fizika, Abs. 7Zh203

79

AUTHOR: Rudina, M.P.

TITLE: Connection between ionospheric absorption of radio waves and magnetic activity

CITED SOURCE: Tr. Sibirsk. fiz.-tekhn. in-ta pri Tomskom un-te,  
vy\*p. 41, 1962, 72-74

TOPIC TAGS: radio wave absorption, ionosphere, anomalous absorption, terrestrial magnetism

TRANSLATION: Data of the measurement of absorption and the behavior of the horizontal component of the earth's magnetic field for 14, 15, and 16 July 1959, as observed in Tomsk, are compared. The results obtained confirm the connection between the anomalous absorption of radiowaves in the ionosphere and the state of the earth's magnetic field in the upper ionosphere.  
Card 1/2

L 16840-63 EWT(d)/EWT(1)/FCC(w)/BDS/EEC-2/ES(v) AFFTC/ASD/AFMDC/ESD-3/  
APGC Pe-4/Pg-4/P1-4/P1-4/Po-4/Pq-4 PT-2/0W S/0058/63/000/007/H030/H031  
ACCESSION NR: AR3006328

90

SOURCE: RZh. Fizika, Abs. 7Zh204

AUTHOR: Rudina, M. P.

TITLE: Absorption of radio waves in the ionosphere and solar activity

CITED SOURCE: Tr. Sibirsk. fiz.-tekhn. in-ta pri Tomskom un-te,  
vy\*p. 41, 1962, 75-79

TOPIC TAGS: radio wave absorption, ionosphere, solar activity,  
sunspot number

TRANSLATION: Measurements were made of the absorption coefficient  
L in the ionosphere at frequencies 2.2 and 3 Mcs, in 1958--1960.  
The course of L over the indicated period is compared with the  
change in the monthly average of the relative number of sun spots W.

Card 1/2

L 16840-63

ACCESSION NR: AR3006328

A correlation analysis shows that L and W are connected during the time of large solar activity. This connection breaks down with decreasing solar activity.

DATE ACQ: 15Aug63

SUB CODE: PH, GE

ENCL: 00

Card 2/2

L 16839-63 EWT(1)/BDS/EEC-2/ES(v) AFFTC/ASD/AFMDC/ESD-3/APGC Pe-4  
Pl-4/Po-4/Pq-4 PT-2/GW

ACCESSION NR: AR3006329

S/0058/63/000/007/H031/H031

78

SOURCE: RZh. Fizika, Abs. 7Zh205

AUTHOR: Rudina, M. P.

TITLE: Reflection coefficient from the F2 layer (Rho) and the parameter Beta

CITED SOURCE: Tr. Sibirs. fiz.-tekhn. in-ta pri Tomskom un-te, vy\*p. 41, 1962, 80-81

TOPIC TAGS: radio wave reflection, ionosphere, reflection coefficient, turbidity coefficient

TRANSLATION: Results are presented of an analysis of 2.2 Mc signals reflected from the ionosphere. It is shown that the reflection coefficient  $\rho$  and the "turbidity coefficient"  $\beta$  are in most cases reciprocally related. The dependence of  $\rho$  and  $\beta$  on the height of re-

Card 1/4

L 19655-63 ENT(1)/BDS/EZC-2/ES(v) AFFTC/ASD/AFMDC/ESD-3/APGC Pe-4/  
Pi-4/Po-4/Pq-4 PT-2/GW s/0058/63/000/008/H032/H032  
ACCESSION NR: AR3007000

SOURCE: RZh. Fizika, Abs. 8Zh208

AUTHOR: Rudina, M. P.; Grokhотова, Б. А.

TITLE: Rate of vertical drift in the F region and number of collisions, as obtained by measuring the reflection coefficient

CITED SOURCE: Tr. Sibirs. fiz.-tekhn. in-ta pri Tomskom un-te, vy\*p. 41, 1962, 92-97

TOPIC TAGS: F region, vertical drift, collision number, ionosphere  
radio sounding, reflection coefficient

TRANSLATION: Results are presented of the analysis of photographs of fluctuating signals of first and second multiplicity, reflected from the F<sub>2</sub> layer in vertical radio sounding of the ionosphere at 2.5--3.5 Mc during February-March 1958. The signals were recorded

Card 1/2

80

L 19655-63

ACCESSION NR: AR3007000

without separating one of the components of the magnetoionic splitting. The analysis has shown the following: 1) in ~50% of the cases the reflection coefficient is  $\rho > 1$ ; 2) values  $\rho > 1$  cannot characterize the degree of absorption of the radio waves in the ionosphere, but can characterize the diffraction by ionization inhomogeneities of the E and F regions, where the reflected signal is produced; 3) the appearance of periodic oscillations in the magnitude of the signal during the periods when the signal increases, when the illumination of the ionosphere decreases, is a more sensitive indicator of the existence of vertical motion than is the variation of  $h_0$ , and can be used to calculate the vertical drift; 4) the frequency of collisions in the F layers, determined from the measured values of the reflection coefficient and from the ionograms agrees with those previously known.

DATE ACQ: 06Sep63

SUB CODE: PH, AS

ENCL: 00

Card 2/2

"APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0

RUDINA, M. P.

"On the Morphology of Radio Wave Absorption in the Lower Ionosphere."

summary to be presented at the 13th Gen Assembly, IUGG, Berkeley, 19-31 Aug 63.

APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0"

L 38557-65 FWT(1)/EWG(v)/FCC/EEC-4/EEC(t)/EWA(h) Po-4/Pa-5/Pq-4/Pae-2/Peb/P1-4

CW

ACCESSION NR: AT5009242

S/2831/64/000/013/0016/0018

44  
B+1

AUTHOR: Rudina, M. P.; Zholdubayeva, G. Kh.

TITLE: Additional ionization in the E-layer

SOURCE: AN SSSR. Mezhdunovostvennyy geofizicheskiy komitet. V razdel programmy  
MGG: Ionosfera. Sbornik statey, no. 13, 1964, 16-18

TOPIC TAGS: ionospheric station, electronic density, critical frequency, ionization

ABSTRACT: Use of an automatic ionospheric station made it possible to determine supplemental reflections between the E- and F1-layers. The maximum electronic density in those layers is characterized by a consistent number of supplemental reflections at heights of 200—250 km. The critical frequency of the maximum density changes from 4—5 Mc. One submaximum is found on the illuminated side, and it is considered as the E2-layer, and the other is observed simultaneously with the reflection from the F1-layer, and it is referred to as F0. The critical frequencies of these layers differ from those of the E-layer. The reflections from E2- and F0-layers are connected with a decrease of ionization in the E- and F1-layers. It is probable that the E2-, F0-, and Es-layers exchange positions. This phenomenon would seem to be caused by vertical translocations of the E-layer.

[EG]

Orig. art. has: 4 figures and 1 table.

Card 1/2

"APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0

RUDINA, M.V., kand. fiziko-matem. nauk

Academy of Sciences of the Kazakh S.S.R. and ionospheric research  
during the International Years of the Quiet Sun. Vest, AN Kazakh,  
SSR 21 no.9:88-89 S '65. (MIRA 18:9)

APPROVED FOR RELEASE: 06/20/2000

CIA-RDP86-00513R001445920015-0"

LIVSHITS, G.Sh.; FESENKO, V.G., akademik, red.; IDLIS, G.M., doktor fiz.-matem.nauk, zamestitel' red.; PYASKOVSKAYA-FESENKOVA, Ye.V., doktor fiz.-matem.nauk, red.; ROZHKOVSKIY, D.A., doktor fiz.-matem., nauk, red. toma; RUDINA, M.P., kand.fiz.-matem.nauk, red.; ROZHKOVSKIY, D.A., doktor fiz.-matem.nauk, red.

[Light scattering in the atmosphere. Pt.1.] Rassieianie sveta v atmosfere. Alma-Ata, Nauka. Pt.1. 1965. 176p (Akademija nauk Kazakhskoi SSR. Astrofizicheskii institut. Trudy, vol.6) (MIRA 18:5)

ZINCHENKO, V.A.; RUDINA, S.I.

Colorimetric determination of titanium in aluminum and magnesium  
by means of diantipyrylmethane. Zav.lab. 27 no.8:956-958 '61.  
(MIRA 14:?)

1. Vsesoyuznyy nauchno-issledovatel'skiy alyuminiyevo-magniyevyy  
institut.  
(Titanium--Analysis) (Aluminum--Analysis) (Magnesium--Analysis)

ACC NR: AP7005675

(A)

SOURCE CODE: UR/0413/67/000/002/0147/0147

INVENTOR: Motovilov, O. A.; Rozhdestvenskiy, V. N.; Rudina, O. G.

ORG: none

TITLE: A method of producing films from a mixture of substances by cathodic sputtering. Class 48, No. 190757

SOURCE: Izobreteniya, promyshlennyye obraztsy, tovarnyye znaki, no. 2, 1967, 147

TOPIC TAGS: thin film, ~~multiple~~<sup>metal</sup> film, film deposition, ~~method~~, cathodic sputtering, ~~method~~ metal deposition, cathode

ABSTRACT: This Author Certificate introduces a method of producing films from mixtures of substances by cathodic sputtering. To obtain films with a given composition and thickness, the film is deposited on the substrate successively from each of the cathodes, which are insulated from one another. The deposition time under each cathode depends on the required thickness of a porous layer from the component of a corresponding cathode. [MS]

SUB CODE: 11,09/ SUBM DATE: none/ ATD PRESS: 5117

Card 1/1

UDC: 621.793.7

55210

26381  
S/032/61/027/008/001/020  
B107/B206

AUTHOR: Zinchenko, V. A., and Rudina, S. I.

TITLE: Colorimetric determination of titanium in aluminum and magnesium by using diantipyryl methane

PERIODICAL: Zavodskaya laboratoriya, v. 27, no. 8, 1961, 956-958

TEXT: A method for the determination of from  $1 \cdot 10^{-4}$  to  $5 \cdot 10^{-5}\%$  of Ti in highly pure aluminum is described. The determination of titanium with diantipyryl methane was proposed by A. A. Minin (Ref. 2: Uchenyye zapiski Permskogo gosudarstvennogo universiteta, v. XI, no. 4, 177(1956)). In acid solution diantipyryl methane forms a bright yellow titanium complex; the reaction is 20 times more sensitive than that with  $H_2O_2$ . Al, Mg, V, X, fluoride, and phosphate do not interfere; with diantipyryl methane, Zn, Cd, Hg form difficultly soluble compounds, and may thus be filtered off; Cr, Ni and Co interfere by their proper color. The interfering Fe<sup>III</sup> is reduced with ascorbic acid. Diantipyryl methane was prepared from antipyrine and formalin according to a formula by V. P. Zhivopistsev

Card 1/3

Colorimetric determination of...

26301  
S/032/61/027/008/001/020  
B107/B206 X

(Ref.3: Zavodskaya laboratoriya, XXVI, 10, 1187 (1960)). The determination may be carried out in sulfuric acid or in the mixture HCl -  $H_2SO_4$ . Between 0.5 and 8 N the acidity does not affect the optical density. Complex formation is completed after 15 minutes; coloring then remains unchanged for a long time. If Fe<sup>III</sup> exists in the solution, 30 to 40 minutes are necessary for a complete reduction by ascorbic acid. The minimum amount of titanium which may thus be determined, is  $1 \cdot 10^{-4}\%$  of Ti for a weighed portion of 1 g of aluminum. The method was tested by adding certain amounts of titanium to the weighed portion of high-purity aluminum. The sensitivity of the method may be increased by precipitating titanium together with ferric hydroxide, manganese(IV)hydroxide, or magnesium hydroxide, and separating it. In this way, even  $5 \cdot 10^{-5}\%$  of Ti may be determined in a weighed portion of 2 g of aluminum. For the determination of titanium in metallic magnesium larger weighed portions (3-5 g) are used, and titanium is separated by coprecipitation with aluminum hydroxide. There are 2 tables and 3 Soviet references.

Card 2/3

Colorimetric determination of...

26381  
S/032/61/027/008/001/020  
B107/B206

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy al'yuminiiyevo-magniyevyy institut (All-Union Scientific Research Institute of Aluminum and Magnesium)

Card 3/3

KAKABADZE, N.A.; SARKISOVA, M.G.; RUDINA, Z.B.

Feldspar-quartz sands and granites of Georgia considered as new-type raw materials for the fine-ceramic industry. Soob. AN Gruz.SSR 18 no.2:167-174 F '57. (MIRA' 10:?)

1. Kavkasskiy institut mineral'nogo syr'ya, Tbilisi. Predstavleno akademikom R.I. Agladze.  
(Georgia--Ceramic materials)

EX  
1931

Proteins and amino acids. III. Synthesis of  $\gamma$ -L-glutamyl-L-tyrosine. F. Sorm and J. Rudinger (Tech. Univ., Prague). Collection Czech. Chem. Commun. 15, 491-500 (1950) [in English]; cf. C.I. 45, 1175d; Le Quesne and Young, C.I. 45, 1956f. N( $\gamma$ -L-Glutamyl)-L-tyrosine has been synthesized by an unequivocal method. The compd. prep'd. by Bergmann and co-workers (C.I. 28, 40750) is shown to be the  $\alpha$ -isomer. The  $\alpha$ -Me ester of carboxy-L-glutamic acid prep'd. by Harrington and Mead (C.I. 29, 72841) is shown to contain the  $\gamma$ -in addition to the  $\alpha$ -isomer. The homogeneity of the compds. was tested by paper chromatography. N-Carbobenzyloxy- $\gamma$ -L-glutamyl hydrazide (I, and V, C.I. 43, 6078g) was converted to the Me ester (II) by  $\text{CH}_2\text{N}_2$ , then by  $\text{BrH}$  to the  $\alpha$ -Me ester of the  $\gamma$ -benzylidene hydrazide of N-carbobenzyloxy-L-glutamic acid, m.p. 123.5-135°.  $\alpha$ -Me ester of N-(N-carbobenzyloxy- $\gamma$ -glutamyl)glycine Et ester, m.p. 80-90°, from I by conversion to the azide and coupling with  $\text{HNCH}_2\text{CO}_2\text{Et}$ . N-( $\gamma$ -L-Glutamyl)-L-tyrosine, m.p. 220-22° (decompn.), was prep'd. from V3 N-carbobenzyloxy- $\gamma$ -L-glutamyl-L-tyrosine with H- and Pd.

10

CA

Lithium aluminum hydride and kindred agents in organic chemistry. Josef Rudinger, Miloslav Ferles, and Miroslav Protiva. *Chem. Listy* 45, 309-714 (1951).—An exhaustive review with 559 references (literature reviewed up to the end of June, 1951). M. Hudlický

RUDINGER, JOSEF ~~REDACTED~~

*Syntheses in the oxytocin field. I. New synthesis of S-benzyl-L-cysteinyl-L-prolyl-L-leucylglycine amide.* Milan Záoral and Josef Rudinger (Czech. akad. věd, Prague), *Zpráva o výzkumu v oblasti chemie (Czechoslov. Chem. Com., 20, 1945, 338)* (in English) of preceding abstract. New syntheses of *L-leucyl-L-leucylglycine* and *L-prolyl-L-leucylglycine* amide are described. The synthesis of the tetrapeptide is shorter by 2 steps than the original synthesis of Du Vigneaud (*J.A. 48, 13311, 49, 6108g*).  $H_3NCH_2CO_2Et$  (0.8 g.) was added at  $-5^\circ$  to a mixed  $CO_2Et$  (0.8 g.) in  $CHCl_3$  was added at  $-5^\circ$  to a mixed anhydride prep'd. from 2 g. carbobenzyloxy-L-leucine (I) and 1.1 g.  $ClCO_2CHMeEt$  in 5 ml.  $CHCl_3$  in the presence of 0.80 g. 1-Et<sub>4</sub>N<sub>2</sub>H, the mixt. was cooled 10 min., allowed to stand at lab. temp. 30 min., the  $CHCl_3$  evapd., the residue dissolved in AcOEt, the solvent evapd., and the residue extd. with 20 ml. H<sub>2</sub>O and 30 ml. AcOEt. The AcOEt layer was washed 3 times with 10% HCl, 3 times with 5%  $NaHCO_3$ , and evapd. to give 2.3 g. *Ei ester* (II) of carbobenzyloxy-L-leucylglycine, m. 105-8° (from AcOEt-petr. ether). Treating 22 g. II with 20 ml. 37% HBr in AcOH, adding the HBr salt, washing the ppt. with Et<sub>2</sub>O dissolving it in  $CHCl_3$ , treating the soln. with  $NH_3$  in  $CHCl_3$ , filtering off the  $NH_3$  Br, evap. the solvent *in vacuo* at 30-5°, and dilg. the residue with  $CHCl_3$  gave *Ei ester* of L-leucylglycine (III). This soln. was mixed with a mixed anhydride prep'd. from L-carbobenzyloxyproline (14.2 g.) and 7.8 g.  $ClCO_2CHMeEt$  in the presence of 0.5 g. 1-Et<sub>4</sub>N<sub>2</sub>H and worked up as above to give 20.2 g. of the *Ei ester* of carbobenzyloxyprolyl-L-leucylglycine (IV), m. 160-1° (from AcOEt-Et<sub>2</sub>O). Decarbobenzyloxylatation of IV could be carried out by catalytic hydrogenation over 5% Pd-C in EtOH in the presence of an equiv. amt. of HCl. The N-carboxy anhydride of L-leucine (850 mg. in 5 ml. AcOH) added at  $-70^\circ$  to  $70^\circ$  to  $57^\circ$  in  $H_3NCH_2CO_2Et$  in 5 ml.  $CHCl_3$ , the mixt. kept at  $-70^\circ$  30 min., heated to room temp., cooled to 0° after the  $CO_2$  evolution ceased, and treated during 30 min. with carbobenzyloxy-L-proline chloride (prep'd. from 1.5 g. carbobenzyloxy-L-proline and 1.3 g. PCl<sub>5</sub>) in  $CHCl_3$  gave 1.1 g. IV (recrystd. from AcOEt-petr. ether). Satg. a soln. of 1.9 g. IV in 20 ml. MeOH with  $NH_3$  at 0°, leaving the mixt. closed 3 days at room temp., distg. off the MeOH, and crystg. the residue from EtOH-petr. ether gave 1.7 g. carbobenzyloxy-L-prolyl-L-leucylglycine amide (V), m. 102-3°. Hydrogenation of 3.2 g. V in 30 ml. EtOH (contg. an equiv. amt. of HCl) over 5% Pd-C, evapn. of the solvent, treatment of the residue with a  $CHCl_3$  soln. of  $NH_3$ , removal of the  $NH_3$  Br, and evapn. of the filtrate *in vacuo* gave 86% L-prolyl-L-leucylglycine amide (VI) hemihydrate, m. 122-3°.

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(from  $H_2O$ ). VI was also obtained by heating 3.4 g. V with 15 ml. 15% HBr in AcOH 10 min. at  $0^\circ$ , pptg. the HBr salt with  $Et_2O$ , reprecip. it with  $Et_2O$  from the EtOH soln., and crystg. from EtOH-Et<sub>2</sub>O gave 2.6 g. VI.HBr, m. 191-2°. Shaking a mixt. obtained by adding the hemihydrate (or HBr salt) of VI in CHCl<sub>3</sub> to a mixed anhydride prep'd. from N-carbobenzyloxy-S-benzyl-L-cysteine (800 mg.), CICO<sub>2</sub>CHMeEt (320 mg.), and 1-EtC<sub>6</sub>H<sub>5</sub>N (270 ml.) in 8 ml. CHCl<sub>3</sub> gave 80% carbobenzyloxy-S-benzyl-L-cysteinyl-L-prolyl-L-leucylglycine (VII), m. 170-1° (from EtOH-H<sub>2</sub>O). Decarbobenzyloxylation of 800 mg. VII by heating 10 min. at  $80^\circ$  with 3 ml. 15% HBr in AcOH, pptn. of the HBr salt with  $Et_2O$ , decantation with  $Et_2O$ , diln. with  $H_2O$ , extn. with AcOEt, treatment of the aq. layer with 600 mg. Ag<sub>2</sub>CO<sub>3</sub>, removal of the Ag with H<sub>2</sub>S, and evapn. of the filtrate after the removal of Ag<sub>2</sub>S gave 470 mg. amorphous hygroscopic S-benzyl-L-cysteinyl-L-prolyl-L-leucylglycnamide (VIII). HBr salt, decompd. above 125°, picrate, aquahydrate, decompd. 105°. Adding 2.0 g. of the Et ester L-leucine in 5 ml. CHCl<sub>3</sub> to a mixed anhydride prep'd. from 4.5 g. carbobenzyloxy-L-proline (m. 79-7°), 2.6 g. CICO<sub>2</sub>CHMeEt, and 2.1 g. 1-EtC<sub>6</sub>H<sub>5</sub>N in 10 ml. CHCl<sub>3</sub> yielded 6 g. Et ester of carbobenzyloxy-L-prolyl-L-leucine in 98° (from AcOEt-petr. ether), hydraside (VIIa), m. 133° (from the same solvent). VIIa (2 g.), 20 ml. 10% HCl, and 10 ml. Et<sub>2</sub>O treated while cooling and stirring with 400 mg. NaNO<sub>2</sub> in 3 ml. H<sub>2</sub>O, seprg. the ether layer, washing with H<sub>2</sub>O, and drying over Na<sub>2</sub>SO<sub>4</sub>.

adding this to 800 mg. benzyl ester of glycine in 10 ml. CHCl<sub>3</sub> gave, after 12 hrs. at room temp., 1.2 g. benzyl ester of carbobenzyloxy-L-prolyl-L-leucylglycine, m. 116-17° (from Me<sub>2</sub>CO-petr. ether). Adding under ice-cooling 6.2 g. 1-EtC<sub>6</sub>H<sub>5</sub>N and tosyl-L-proline chloride (prep'd. from 5.2 g. tosyl-L-proline and 3.7 g. PCl<sub>5</sub>) in CHCl<sub>3</sub> to the Et ester of L-leucylglycine-HCl (prep'd. by hydrogenation of 6 g. of the Et ester of carbobenzyloxy-L-leucylglycine) in CHCl<sub>3</sub>, stirring the mixt. 30 min., distg. off the solvent, and extg. the residue with AcOH yielded 0.9 g. Et ester of tosyl-L-prolyl-L-leucylglycine, m. 145-6° (from AcOEt-petr. ether). R<sub>f</sub> values for VI and VIII in BuOH-AcOH-H<sub>2</sub>O were 0.55 and 0.73, resp. II. Syntheses of L-cysteinyl-L-tyrosyl-glycine, L-cysteinyl-L-tyrosyl-L-leucine, and L-cysteinyl-L-tyrosyl-L-isoleucine derivatives. Jan Honzík and Josef Rudinger. *Chem. Listy* 49, 751-8; *Collection Czechoslov. Chem. Commun.* 20, 1100-R (1955) (in English).—Dissolving 21.1 g. S-benzyl-L-cysteine in 75 ml. 2N NaOH, adding 30 ml. Me<sub>2</sub>CO, and, simultaneously, with cooling and stirring, a soln. of 20 g. p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>Cl in 60 ml. Me<sub>2</sub>CO and 26 ml. 4N NaOH, stirring the mixt. 1 hr., neutralizing the mixt., distg. the Me<sub>2</sub>CO *in vacuo*, and acidifying the mixt. with HCl gave an oil which crystallized after standing to give 20-25.5 g. tosyl-S-benzyl-L-cysteine (I), m. 125-6° (from C<sub>6</sub>H<sub>6</sub>); acid chloride (from SOCl<sub>2</sub> in 70-80% yield), m. 113-14° (from CHCl<sub>3</sub>-petr. ether). Adding 5.7 g. of the chloride of I in AcOEt to a cooled satd. soln. of 7.3 g. Et ester of L-tyrosine (from AcOEt, washing the AcOEt soln. with dil. HCl, H<sub>2</sub>O, N<sub>2</sub>CO<sub>3</sub>, and H<sub>2</sub>O, and evapn. the solvent *in vacuo* gave, at + crystn. from C<sub>6</sub>H<sub>6</sub>, 7.6 g. Et ester of tosyl-S-benzyl-L-

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cysteinyl-L-tyrosine (II), m. 115-18° (from AcOEt-petr. ether). Sapon. of the Et ester of II with 4N NaOH at room temp. gave 86% II, m. 160-8° (from aq. EtOH); hydrazide (from the ester and 80%  $N_2H_4 \cdot H_2O$  in EtOH), m. 201° (from aq. EtOH). Dissolving 0.3 g. of the hydrazide of II in a mixt. of 3 ml. N HCl and 5 ml. AcOH, adding 14 ml. Et<sub>2</sub>O, and, in a special app., allowing to work at -18°, 0.102 g. NaNO<sub>2</sub> in 1 ml. H<sub>2</sub>O, stirring 1 min., adding 80 ml. 28.5% satd. NaCl soln. cooled to -18° and 18 ml. Et<sub>2</sub>O of the same temp., stirring the mixt. intensively for 0.5 min., draining the aq. layer and washing the org. layer twice with satd. NaCl soln. at -18° (the whole operation has to be finished in 7 min.) transferring the ether soln. of the azide into a precooled flask, drying it with Na<sub>2</sub>SO<sub>4</sub>, adding to a precooled soln. of 0.17 g. freshly distd. Et ester of glycine (III) in 3 ml. AcOEt, and allowing the mixt. to stand at 0° overnight gave an oil which crystl. The ether soln. and the crystals were separately worked up with dil. HCl, H<sub>2</sub>O, 5% NaHCO<sub>3</sub>, and H<sub>2</sub>O, the solvent evapd., and the crystals recrystd. from C<sub>6</sub>H<sub>6</sub> to give 0.63 g. Et ester of tosyl-S-benzyl-L-cysteinyl-L-tyrosylglycine (IV), m. 134.5° (from aq. EtOH). Treating a suspension of 2.5 g. HCl salt of O-acetyl-L-tyrosine in 50 ml. tetrahydrofuran at 40-50° with COCl<sub>2</sub>, 3.5 hrs., passing dry air through the soln. 2 hrs., distg. off the solvent *in vacuo*, and dissolving the residue

in Et<sub>2</sub>O gave on standing at 0°, 2 g. L-4-(*p*-acetoxybenzyl)-azazolidin-2,5-dione (V), m. 118-20° (from AcOEt-petr. ether). Dissolving 0.2 g. III and 0.8 ml. EtNC<sub>6</sub>H<sub>5</sub> in 20 ml. AcOEt, cooling the mixt. to -60°, adding a soln. of 0.5 g. V in 10 ml. AcOEt, shaking the mixt. occasionally at -60°, heating up to room temp., cooling to -18°, treating with 0.7 g. of the chloride of I in 10 ml. AcOEt, heating up to room temp., washing with dil. HCl, H<sub>2</sub>O, 5% NaHCO<sub>3</sub>, H<sub>2</sub>O, drying with Na<sub>2</sub>SO<sub>4</sub>, evapg. the solvent, and crystg. the residue from AcOEt and petr. ether gave 0.57 g. Et ester of tosyl-S-benzyl-L-cysteinyl-(O-acetyl-L-tyrosyl)glycine (VI), m. 184-5° (from aq. EtOH). Hydrolysis of the ester of IV (0.2 g.) with 0.4 ml. 4N NaOH and 0.6 ml. H<sub>2</sub>O 45 min. at room temp. gave 0.11 g. IV (hydrate), m. 125° (from aq. EtOH). The same compd. IV was obtained in 50% yield by hydrolyzing 0.13 g. of the Et ester of VI with 0.17 ml. 4N NaOH. Hydrazide (VII) of IV, prep'd. in 90-3% yields from esters of IV and V with 80%  $N_2H_4 \cdot H_2O$ , m. 217-18° (from aq. EtOH). VII (0.8 g.) and 0.35 g. Et ester of L-leucine (VIII) gave 0.66 g. Et ester of tosyl-S-benzyl-L-cysteinyl-L-tyrosyl-L-leucine (IX), m. 184° (from aq. EtOH). Treating 2 g. of the hydrazide of II in 14 ml. AcOH, 4 ml. N HCl, 3 ml. H<sub>2</sub>O and 36 ml. Et<sub>2</sub>O with 0.3 g. NaNO<sub>2</sub> in 2 ml. H<sub>2</sub>O in the manner described above, and adding the soln. of the azide to a soln. of 0.63 g. of the Me ester of leucine in AcOEt gave 1.53 g. Me ester of IX, m. 193° (from aq. MeOH). V (1.84 g.), 1.18 g. VIII, and 2.35 g. acid chloride of I gave 0.4 g. Et ester of tosyl-S-benzyl-L-cysteinyl-(O-acetyl-L-tyrosyl)-L-leucine, m. 160-8° (from iso-PrOH). Sapon. of this ester with 4N NaOH (45 min. at

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room temp.) gave 88% IX.<sup>1/2</sup>H<sub>2</sub>O, m. 93-101°. The same compd. IX was obtained from the Et and Me esters of IX. Hydraside of IX, m. 227°, was prep'd. in 30% yield by refluxing the Me ester with 80% N<sub>2</sub>H<sub>4</sub>H<sub>2</sub>O. Hydraside (1.8 g.) of II, 0.22 g. NaNO<sub>2</sub>, and 0.49 g. Me ester of L-isoleucine gave 33-86% Me ester of *tosyl-S-benyl-L-cysteinyl-L-tyrosyl-L-isoleucine* (X), m. 193-5° (from aq. MeOH). Sapon. of this ester gave 50% X, m. 119-23°. Hydraside of X, prep'd. from the Me ester and abs. N<sub>2</sub>H<sub>4</sub> in 7 days at room temp., 80% yield, m. 242-3° (from aq. EtOH). Transforming the hydraside of IX (0.83 g.) in 2 ml. N HCl, 3 ml. H<sub>2</sub>O, 8 ml. AcOH, and 15 ml. Et<sub>2</sub>O with 0.06 g. NaNO<sub>2</sub> in 0.5 ml. H<sub>2</sub>O to the azide, and adding the azide soln. to 0.1 g. III in AcOEt gave 0.28 g. Et ester of *tosyl-S-benyl-L-cysteinyl-L-tyrosyl-L-leucylglycine* (XI), m. 194-5° (from 75% EtOH). The same compd. was prep'd. by adding a mixt. obtained from 0.06 g. III, 1 ml. HOP-(OEt)<sub>2</sub>, and 0.16 ml. Et pyrophosphite to 0.34 g. IX (obtained by azeotropic dehydration of its hemihydrate), by heating the mixt. 30 min. at 80-90°, pouring into 60 ml. H<sub>2</sub>O, and allowing to stand in the icebox overnight. Washing the ppt. with H<sub>2</sub>O, hot 5% NaHCO<sub>3</sub>, dissolving it in hot EtOH, pottg. with H<sub>2</sub>O, and recrystg. from aq. EtOH gave 0.2 g. of the Et ester of XI.

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Amino acids and peptides. VIII. Peptides of 2,6-di-  
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Lithium aluminum hydride and kindred agents in organic chemistry. III. Miroslav Ferles and Josef Rudinger (Tech. Univ., Prague, Czech.). Chem. Abstr. 48, 1463 (1953).—A supplement to an earlier review (C.A. 46, 44634) involving literature from July 1951 to June 1952. The review contains 413 references and tables of all compds. reduced by LiAlH<sub>4</sub>-type agents. M. Hudlicky

## CZECH

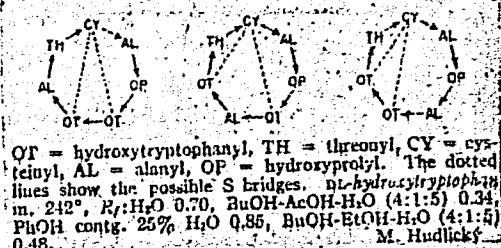
Amino acids and peptides. VIII. Peptides of 2,4-diaminobutyric acid. [Milan Zaoral, Josef Rudinger, and František Šorm (Czech. Akad. Věd, Prague, Czech.). *Chem. Listy* 47, 427-39 (1953); cf. *C.A.* 48, 3903e.—[In this abstr., R = carbobenzoyloxy throughout.] Several peptides and intermediates of L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H (I) were prep. L-Glutamic acid gave by the Schmidt degradation 2% I; di-HCl salt, m. 105-8°; dipicrate, m. 181°; 2-naphthalenesulfonate, Cu<sub>11</sub>H<sub>2</sub>N<sub>2</sub>O<sub>8</sub>S<sub>2</sub>2H<sub>2</sub>O (the formula in the original is wrong), m. 215° (from H<sub>2</sub>O). L-RNHCH<sub>2</sub>CH<sub>2</sub>CH(NHR)CO<sub>2</sub>H (II), m. 90-1°; hydrazide, m. 160-2°, prep'd. through II Et ester or by treating 1-carbobenzoyloxy-3-carbobenzoyloylaminoo-2-pyrrolidinone (IIA) with 80% NaH<sub>2</sub>O soln. in EtOH. L<sub>2</sub>HCl (16 g.) in 240 ml. N NaOH with 12 g. ClCO<sub>2</sub>CH<sub>2</sub>Ph (III) gave 7.6 g. (24.8%) II, m. 90-2°, 8.4 g. (39.8%) RNHCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H (IV), m. 231° (from H<sub>2</sub>O), and 3.7 g. (23.1%) I. Similarly, 3 g. H<sub>2</sub>N(CH<sub>2</sub>)<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H.HCl in 47 ml. N NaOH and 2.8 g. II in 32 ml. N NaOH yielded 1.6 g. (34.5%) L-RNH(CH<sub>2</sub>)<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H, m. 255° (from H<sub>2</sub>O). HCl salt of IV Et ester (65.5% from IV with 8% HCl in EtOH), m. 152-3°. II (4 g.) was transformed into 3.3 g. (86.5%) IIA, m. 113-14° (from AcOEt-petr. ether), which in turn was converted to 57.3% amide of II, m. 107° (from aq. AcOH). IV with COCl<sub>2</sub> in dioxane gave L-L-(2-carbobenzoylaminomethyl)-2,5-oxazolidinedione. The hydrazide of II (1.8 g.) in 20 ml. AcOH treated with 10 ml. HCl (1:10), 50 ml. Et<sub>2</sub>O, and, with cooling and stirring, 330 mg. NaNO<sub>2</sub> in 3 ml. H<sub>2</sub>O, the mixt. poured into 50 ml. ice water, the ether layer washed with H<sub>2</sub>O to remove the mineral acid, the dried ether soln.

added dropwise to H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>Et in CHCl<sub>3</sub>, the mixt. let stand overnight, the solvents distd. off at 40° *in vacuo*, the residue dissolved in AcOEt, the soln. washed 3 times with HCl (1:5), 3 times with a satd. soln. of Na<sub>2</sub>CO<sub>3</sub>, and finally twice with 10% Na<sub>2</sub>CO<sub>3</sub> soln., dried, evapd. *in vacuo*, and the residue crystall. from aq. EtOH gave 1.6 g. (70.8%) L-RNHCH<sub>2</sub>CH<sub>2</sub>CH(NHR)CONHCH<sub>2</sub>CO<sub>2</sub>Et (V), m. 130-1°, also obtained (75%) by heating 0.4 g. IIA with 0.5 ml. H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>Et 5 min. at 135°. V is hydrolyzed with N NaOH to 79.5% free acid, m. 142-3°. V (500 mg.) in 5 ml. AcOH heated at 60° with a soln. prep'd. by mixing 3.1 g. 50% HI with 5.2 g. Ac<sub>2</sub>O, the PhCl<sub>2</sub>I removed by Et<sub>2</sub>O extn., the iodine pptd. from the aq. layer as PbI<sub>2</sub>, the Pb ions removed with H<sub>2</sub>S, and the soln. evapd. several times to dryness, yielded 104 mg. (98.2%) L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CONH-CH<sub>2</sub>CO<sub>2</sub>H, dipicrolonate monohydrate, m. 202-3° (from aq. EtOH). II (1.8 g.) and L-BuCH(NH<sub>2</sub>)CO<sub>2</sub>Et (from 1 g. of the HCl salt) gave by the azide method 1.95 g. (82%) L-RNHCH<sub>2</sub>CH<sub>2</sub>CH(NHR)CONHCHBuCO<sub>2</sub>Et-L (VII), also prep'd. by the anhydride method by treating at -5° 2 g. II in 20 ml. CHCl<sub>3</sub>, 0.9 g. III, and 0.6 g. MeNC<sub>4</sub>H<sub>9</sub> with CHCl<sub>3</sub> soln. of L-BuCH(NH<sub>2</sub>)CO<sub>2</sub>Et (from 1 g. HCl salt), letting the mixt. stand 14 hrs., and evapg. it *in vacuo* to give 1.05 g. (40%) VII, m. 108°; the free acid, m. 155° (from aq. EtOH), treated with HI, gave 88% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CONHCHBuCO<sub>2</sub>H (dipicrolonate, m. 225-7° (from H<sub>2</sub>O)). L-RNHCH<sub>2</sub>CH<sub>2</sub>CH(NHR)CONHCH(CO<sub>2</sub>E)CH<sub>2</sub>CO<sub>2</sub>Et-L, m. 130-1° (from aq. EtOH), prep'd. in 70.1% yield by the anhydride method from 4 g. II, 1.75 g. di-Et glutamate-HCl, 1.8 g. III, and 1 g. MeNC<sub>4</sub>H<sub>9</sub>, gave by

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<sup>21</sup> dicarbobenzyloxydipeptide, m. 189-90° (from aq. EtOH), from which was obtained 91.2% L-2,4-diaminobutyryl-L-glutamic acid monohydrate, m. 192-3° (from aq. EtOH). L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHR)CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>)CO<sub>2</sub>Et, m. 140-2° (from aq. EtOH), prep'd. in 01.8 g. yield by the azide method and in 50.2% yield by the anhydride method, was hydrolyzed quantitatively to the free acid, m. 100-2° (from aq. EtOH), which, hydrogenated in aq. AcOH over 15% Pd-C, yielded 75% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NH<sub>2</sub>)CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>)CO<sub>2</sub>H; triphloranol dihydrate, m. 232° (from H<sub>2</sub>O); triphanol, m. 228-7° (from H<sub>2</sub>O). The monocryst. L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHR-L)CO<sub>2</sub>Et (1.7 g.), prep'd. by the anhydride method) was hydrolyzed to 4.7 g., prep'd. in 44.5% yield, m. 198-9° (from aq. AcOH). El ester, m. 143-3° (from aq. EtOH), which, hydrogenated in AcOH over Pd-C, gave 91% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NH<sub>2</sub>)CO<sub>2</sub>H, trypanate, m. 213-16° (decompn.) (from H<sub>2</sub>O). RNIICH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHR)Bu-L)CO<sub>2</sub>Et, m. 68-9° (from aq. EtOH), prep'd. in 63% yield by the anhydride method and in 33% yield by the azide synthesis, was sapon'd. to 74% free acid, m. 143-5° (from aq. EtOH), which with HI gave 87.5% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NH<sub>2</sub>)Bu-L)CCl<sub>4</sub>H[bi-(2-naphthalenesulfonate)], m. 190-1° (from H<sub>2</sub>O). RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHR-L)CO<sub>2</sub>Et, m. 153° (from aq. EtOH), prep'd. in CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Et-L)CO<sub>2</sub>Et, m. 153° (from aq. EtOH), prep'd. in 84.5% free acid, m. 177-8° (25.5% yield), was sapon'd. to L-H<sub>2</sub>NCH<sub>2</sub> (from aq. EtOH), and decarbobenzyloxylated to L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NH<sub>2</sub>)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, decompn., m. 193-5°. It was transformed with N<sub>2</sub>H<sub>4</sub>, H<sub>2</sub>O to 77% L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NH<sub>2</sub>)CONHCH<sub>2</sub>CONHNH<sub>2</sub>, m. 178-9° (from H<sub>2</sub>O).

(aq. AcOH), which, by the azide synthesis, yielded 80.5% L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHR)CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>)CO<sub>2</sub>Et, m. 151-3° (from AcOBt-petr. ether). This was transformed to 92% corresponding hydrate, m. 202-3° (from aq. AcOH), which with H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph, HCl gave 41.5% L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHCOCH(NH<sub>2</sub>)CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>)CO<sub>2</sub>Et, m. 203-5° (from aq. AcOH). Decarbobenzyloxylation by hydrogenation in aq. AcOH on Pd-C, liberated 78% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NH<sub>2</sub>)CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>-L)CO<sub>2</sub>Et, m. 215-17° (from H<sub>2</sub>O), IV (4 g.) in 10 ml. CHCl<sub>3</sub> sulfate, m. 215-17° (from H<sub>2</sub>O), IV (4 g.) in 10 ml. CHCl<sub>3</sub> treated at -65° with H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph (from 3.2 g. of its HCl salt), and 1.0 g. MeNC<sub>6</sub>H<sub>5</sub> in 5 ml. CHCl<sub>3</sub>, and finally with  $\rho$ -C<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>NCH<sub>2</sub>COCl (from 3.2 g. C<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>Et) in 15 ml. CHCl<sub>3</sub> gave L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NH<sub>2</sub>)CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>-L)CONHCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph, m. 230-1° (from aq. AcOH); El ester, prep'd. similarly in 44.5% yield, m. 198-9° (from aq. AcOH). IX. Constitution of phalloidins. 2. Bedřich Meloun, Boživoj Keil, and František Šorm, Czech. Akad. Věd, Prague, Czech., *Chem. Listy* 47, 150-10 (1953); cf. *C.A.* 47, 32376. —New expts. with phalloidin show the sequence of L-threonyl-L-cysteinyl-L-alanyl-L-alohydroxyprolyl in the mol. The following possibilities for the structure of phalloidin are suggested:



RUDINGER, Josef

*Amino acids and peptides. X. Some derivatives and reactions of 1-p-toluenesulfonyl-L-pyrrolidin-5-one-2-carboxylic acid.* Jozef Rudinger (Czech. Acad. vuf, Prague, Czech.), *Chem. Listy* 48, 235-43; *Collection Czechoslov. Chem. Commun.* 19, 365-74 (1954) (in English); cf. preceding abstr.—A new method of prepn. of 1-p-toluenesulfonyl-L-pyrrolidin-5-one-2-carboxylic acid (I) is described. The lactam grouping of I is aminated to give  $\gamma$ -amides of  $\alpha$ -MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>NHCH(CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H (II), whereas the  $\alpha$ -amides of II can be obtained by alk. hydrolysis of the corresponding amides of I. I and MeONa give the  $\gamma$ -Me ester of II, L-HO<sub>2</sub>CCH(NH<sub>2</sub>)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H (147 g.) in 500 ml. 4N NaOH treated with cooling with 190 g.  $\rho$ -MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>Cl in 500 ml. Me<sub>2</sub>CO and, at the same time, with 250 ml. 4N NaOCl, the Me<sub>2</sub>CO distd. off, and the soln. acidified to Congo red with HCl gave 264 g. crude II, yielding on recrystn. 208 g. (99%) II, m. 145-6° (from H<sub>2</sub>O). Ether extn. of the mother liquor yielded 27 g. to make the total yield 78%. A small amt. of II di-Et ester, m. 76-8°, was obtained as a by-product. II with Ac<sub>2</sub>O gave the hydrate of I, m. 68-70° (from aq. EtOH); anhyd. I, m. 130°, was also prepnd. by shaking 30.1 g. II with 50 ml. SOCl<sub>2</sub>, 6 hrs., dilg. the mixt. with 50 ml. Et<sub>2</sub>O, filtering, dilg. with 50 ml. Et<sub>2</sub>O, and pptg. with 250 ml. petr. ether (yield 26.1 g. (92%)). Refluxing 30.1 g. II with 50 ml. SOCl<sub>2</sub> gave, after pptg. the product from Et<sub>2</sub>O with petr. ether, 28.1 g. (93%) acid chloride (IIa) of I, m. 83-5° (from Et<sub>2</sub>O). From IIa were prepnd. the following derivs. of I: amide, m. 191-5°; Et ester, m. 115-18° (from EtOH-Et<sub>2</sub>O). From I were prepnd. the following derivs. of I: NH<sub>2</sub> salt, m. 202-4° (from aq. EtOH); MeNC<sub>6</sub>H<sub>4</sub> salt, m. 170° (from EtOH); PhNH<sub>2</sub> salt (C<sub>6</sub>H<sub>5</sub>N<sub>2</sub>O<sub>2</sub>S), m. 142-4°; C<sub>6</sub>H<sub>5</sub>N salt (C<sub>6</sub>H<sub>5</sub>N<sub>2</sub>O<sub>2</sub>S), m. 111°. The treatment of 3 g. II with 2.7 ml. MeNC<sub>6</sub>H<sub>4</sub> and 10 ml. 13% soln. of COCl<sub>2</sub> in PhMe, evapn. of the mixt. *in vacuo*, and washing the residue with H<sub>2</sub>O gave 1.5 g. MeNC<sub>6</sub>H<sub>4</sub> salt of II, m. 170°. I with AcCl or Ac<sub>2</sub>O gave the mixed

anhydride (III) of I and AcOH. III (1.6 g.) in 10 ml. CHCl<sub>3</sub> and 20 ml. 2.5% NH<sub>3</sub> in CHCl<sub>3</sub> gave 0.85 g. NH<sub>4</sub> salt of I, m. 202-4°, and 0.23 g. of the anide of I, m. 192-4°. III (0.85 g.) and PhNH<sub>2</sub> in C<sub>6</sub>H<sub>6</sub> gave 0.35 g. PhNH<sub>2</sub> salt of I, and 0.1 g. I. The chloride of I (0.3 g.) and 0.5 ml. PhNH<sub>2</sub> in 2 ml. CHCl<sub>3</sub> gave 0.38 g. (88%) of the anide (IV) of I, m. 230-2° (from 90% AcOH). Treating 0.9 g. IV in 5 ml. EtOH with 1.25 ml. 4N NaOH, dilg. the mixt. with 50 ml. H<sub>2</sub>O, acidifying, seprg. the ppt., suspending it in 5% NaHCO<sub>3</sub>, removing the undissolved portion (ester amide), and reproto. with acid gave 0.65 g. of the  $\alpha$ -amide of II, m. 188-200°. The ester gave by hydrolysis an addnl. 0.21 g. (0.8 g., 92% total). Treating 0.9 g. IV in 5 ml. MeOH with 5 ml. N MeONa 5 min. and pouring the mixt. into 50 ml. 5% HCl gave 0.97 g. (99%)  $\gamma$ -Me ester  $\alpha$ -amide of II, m. 138° (from aq. MeOH). Similarly was prepnd., from the amide of I, 71%  $\gamma$ -Me ester  $\alpha$ -amide of II, m. 184-5° (from aq. MeOH). Pouring a soln. of 2.8 g. I in 20 ml. N MeONa after 30 min. into 100 ml. 5% HCl gave 2.17 g. (69%)  $\gamma$ -Me ester of II, m. 113-14° (from aq. MeOH). Anhyd. I (14 g.) boiled 15 min. with 50 ml. concd. aq. NH<sub>3</sub> and the mixt. acidified with HCl gave 13.3-14.8 g. (89-98%)  $\beta$ -MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>NHCH(CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CONH<sub>2</sub>, m. 160° (from H<sub>2</sub>O, aq. EtOH, Ac<sub>2</sub>O, or petr. ether). The same compd. was obtained by tosylation of L-glutamine. Boiling a mixt. of 1.41 g. anhyd. I 5 min. with 5 ml. PhNH<sub>2</sub> and pouring the mixt. into 50 ml. 10% HCl gave 1.25 g. (67%)  $\gamma$ -anilide of II, m. 217-18° (from 80% EtOH), and the dianilide, m. 232° (insol. in 5% NaHCO<sub>3</sub>). The  $\gamma$ -anilide of II was also prepnd. in dioxane soln. (40% yield). Heating 1 g. hydrated I 2 min. at 120° with 2 ml. C<sub>6</sub>H<sub>5</sub>N gave 1.1 g. (83%) piperazine of II, m. 88-70° (dihydrate). Refluxing

(over)

2.8 g. anhyd. I with 10 ml. 45%  $\text{NaH}_2$ , yielded 2.8 g. (89%)  $\gamma$ -hydroxide (V) of II, m. 207° (from  $\text{H}_2\text{O}$ ). Treating 3.16 g. V in 20 ml. 3% HCl with 8.5 ml. 10%  $\text{NaNO}_4$ , extg. the oil with ether, evapg. the ether *in vacuo*, treating the residue with 7 ml.  $\text{PhCH}_2\text{OH}$ , heating the mixt. 1 hr. at 100°, and dig. the cooled mixt. with 40 ml.  $\text{Et}_2\text{O}$  gave 0.65 g. (25%) white crystals of the lactam of *N*-*p*-toluenesulfonyl-*L*- $\alpha$ , $\gamma$ -diaminobutyric acid lactam(3-*p*-toluenesulfonyl-2-pyrrolidinone). The filtrate extd. with  $\text{NaHCO}_3$  yielded, after acidification, 1.5 g. (37%) *N*-*p*-toluenesulfonyl-*N*<sub>7</sub>-carbobenzyloxy-*L*- $\alpha$ , $\gamma$ -diaminobutyric acid, m. 140-50°. XI.

A few syntheses of peptides of glutamic acid. *Ibid.* 48, 244-53; *Collection Czechoslov. Chem. Commun.* 19, 375-85 (1951) (in English).—A series of  $\alpha$ - and  $\gamma$ -glutamyl-peptides and glutaminyl peptides has been prep'd. starting with *N*-*p*-toluenesulfonyl-5-pyrrolidone-2-carboxylic acid (I). The isolation of the products after the removal of the tosyl group with Na in  $\text{NH}_3$  was carried out by means of ion exchangers. Anhyd. I (14.15 g.), 7 g.  $\text{H}_2\text{NCH}_2\text{CO}_2\text{Et}$ , 10 ml. HCl, 75 ml. MeCN, and 15 ml.  $\text{EtNC}_2\text{H}_2$  refluxed 3 hrs., the mixt. cooled, the solvent distd. off *in vacuo*, the residue triturated with 50 ml. dil. HCl, and the ppt. purified by dissolving in  $\text{NaHCO}_3$  (5% aq. soln.), and repp'tg. with HCl yielded 15.4 g. (80%) of the *E* ester of *N*-*(p*-toluenesulfonyl- $\gamma$ -L-glutamyl)glycine (II), 190-1°; the same yield was obtained with dioxane as solvent. A compd. insol. in 5%  $\text{NaHCO}_3$ , m. 148° (from 75% EtOH), was isolated as a by-product. Treating 7.7 g. II in 15 ml.  $\text{H}_2\text{O}$  with 15 ml. 4N NaOH, filtering the soln. with C after 20 min., and acidifying the filtrate with 7 ml. HCl gave 5.85 g. (82%) *N*-*(p*-toluenesulfonyl- $\gamma$ -L-glutamyl)glycine, m. 171-2° (from  $\text{H}_2\text{O}$ ). Treating 1.93 g. II in 10 ml. MeOH satd. with  $\text{NH}_3$  yielded 1.81 g. (90%) *N*-*(p*-toluenesulfonyl- $\gamma$ -L-glutamyl)glycynamide (IIIa), m. 208° (from  $\text{H}_2\text{O}$  with EtOH added). Refluxing 1.41 g. I, 1.25 g.  $\text{p-HOC}_2\text{H}_2\text{CH}(\text{NH}_2)\text{CO}_2\text{Et}$ , 10 ml. HCl (III), and 1.25 g.  $\text{EtNC}_2\text{H}_2$  5 hrs. in 10 ml. dioxane, evapg. the solvent *in vacuo*, dissolving and acidifying the residue gave 1.16 g. (47%) *E* ester of *N*-*(p*-toluenesulfonyl- $\gamma$ -L-glutamyl)-*L*-tyrosine

(IV), m. 181-2° (from aq. EtOH); also obtained in 83% yield by heating 10 min. at 130-5° the mixt. of 1.41 g. I with 2.6 g.  $\text{p-HOC}_2\text{H}_2\text{CH}(\text{NH}_2)\text{CO}_2\text{Et}$ . Hydrolysis of 1.31 g. IV with 5 ml. 2N NaOH gave 1.07 g. (80%) hydrate of *N*-*(p*-toluenesulfonyl- $\gamma$ -L-glutamyl)-*L*-tyrosine (V), m. 115-25°, obtained directly by refluxing 7 g. I, 0.3 g. III, and 7 g.  $\text{EtNC}_2\text{H}_2$  5 hrs. in 75 ml. dioxane, evapg. the solvent *in vacuo*, dissolving the residue in  $\text{H}_2\text{O}$ , filtering, acidifying the soln., and saponif. the crude IV (8.7 g.) by shaking 20 min. with 38 ml. 2N NaOH. Th. oil gave 4.5 g. (37%, based on I) V, m. 120-30° (from  $\text{H}_2\text{O}$  with EtOH added). Anhyd. V, a glassy mass, was obtained by heating the hydrate of V 24 hrs. at 100°/0.2 mm. over  $\text{P}_2\text{O}_5$ . Shaking 15.1 g. of the chloride of I in 100 ml.  $\text{CHCl}_3$  10 min. with 7.7 g.  $\text{H}_2\text{NCH}_2\text{CO}_2\text{Et}$ , HCl, 9 g.  $\text{NaHCO}_3$ , and 40 ml.  $\text{H}_2\text{O}$ , seprg. the  $\text{CHCl}_3$  layer, washing it twice with 50 ml. dil. HCl (1:3), evapg. the solvent, and crystg. the residue from 96% EtOH gave 15.55 g. (85%) *E* ester of *N*-*(p*-toluenesulfonyl-*L*-pyrrolidin-5-one-2-carbonyl)glycile (VI), m. 132-3°. Similarly, 8.4 g. of the chloride of I, 25 ml.  $\text{CHCl}_3$ , 5.8 g.  $\text{H}_2\text{NCH}_2\text{CO}_2\text{CH}_2\text{Ph}$ , HCl, 4.8 g.  $\text{NaHCO}_3$ , and 20 ml.  $\text{H}_2\text{O}$  yielded 9.4 g. (78%) of the benzyl ester (VIa), m. 90-2°. VI (3.65 g.) dissolved in 5 ml. 2N NaOEt and filtered after 15 min. into 50 ml. 5% HCl gave 53% *di-E* ester of *N*-*(p*-toluenesulfonyl- $\alpha$ -L-glutamyl)glycine (VII), m. 109°; the *di-Me* ester (VIa), m. 99-100° (from aq. MeOH), was obtained in 44% yield by filtering the soln. of 3.65 g. *di-E* ester of VII in 10 ml.  $\text{NaOMe}$  into 50 ml. 5% HCl. Hydrogenation of 10.30 g. VII in 20 ml. AcOH, 50 ml.  $\text{H}_2\text{O}$ , and 50 ml. 90% EtOH over 1 g. 5% Pd on C yielded 7.6 g. (94%) *N*-*(p*-toluenesulfonyl-*L*-pyrrolidin-5-one-2-carbonyl)glycine (VIII), m. 198-200° (from aq. EtOH). VIII (3.4 g.) dissolved in 7.5 ml. 4N NaOH and acidified, after 10 min. with HCl gave 3.25 g. (91%) *N*-*(p*-toluenesulfonyl- $\alpha$ -L-glutamyl)glycine, m. 208-12°. The same product (m. 212° and 217°) was prep'd. in 92% yield by the hydrolysis of VII and VIIa, by the alk. and acidic hydrolysis of VI (72% and 91%, resp.), and by direct synthesis from 3.01 g. chloride of I

in 20 ml. Et<sub>2</sub>O and 0.75 g. H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>H in 15 ml. 2N NaOH (yield 1.58 g., 43%). N-p-Toluenesulfonyl-L-glutamic acid (7.5 g.) refluxed with 25 ml. SOCl<sub>2</sub>, the chloride treated in 25 ml. CHCl<sub>3</sub> with 6.1 g. III, 25 ml. H<sub>2</sub>O, and 4.2 g. NaHCO<sub>3</sub> until the evolution of CO<sub>2</sub> ceased, and the ppt. filtered and washed with 95% EtOH gave 83% (10.1 g.). Et ester and washed with 95% EtOH gave 83% (10.1 g.). Et ester of N-(p-toluenesulfonyl-L-pyrrolidin-5-one-2-carbonyl)-L-

tyrosine (IX), m. 133° (hydrate) (from 90% EtOH). Treating IX (6.4 g.) with 15 ml. 4N NaOH and acidifying the soln. yielded 5.5 g. (92%) N-(p-toluenesulfonyl-a-L-glutamyl)-L-tyrosine (IXa), m. 213-19°. Refluxing 8 g. VIII 15 min. with 25 ml. concd. aq. NH<sub>3</sub> and acidifying the soln. yielded N-(p-toluenesulfonyl-L-glutamyl)glycine (X), m. 212-13° (from H<sub>2</sub>O). N-p-Toluenesulfonyl-L-glutamine (Xa) (cf. preceding part) (3 g.) in 100 ml. liquid NH<sub>3</sub> treated with 1.3-1.6 g. Na, then with NH<sub>4</sub>OAc until the blue color disappeared, the NH<sub>4</sub> evapd., the residue dissolved in 20 ml. H<sub>2</sub>O and ice, treated with 10 g. NH<sub>3</sub> salt of ion exchanger (Amberlite IRC-50), the ion-exchanger filtered off after 30 min., washed with H<sub>2</sub>O, and the turbid filtrate evapd. at 40° in vacuo to 15 ml., the cooled soln. treated with 1M Ba(OAc)<sub>2</sub> until no ppt. was formed (5-10 ml.), filtered, the filtrate evapd. in vacuo to 10-30 ml., filtered through a column contg. 20 g. Amberlite (in NH<sub>3</sub> cycle), the column washed with distd. H<sub>2</sub>O, and the eluate (150 ml.) evapd. at 40° in vacuo to 20 ml. and treated with EtOH gave 1.25 g. (86%) L-glutamine, m. 188°, [α]<sub>D</sub><sup>25</sup> 8.9°. The same product was prep'd. by the reduction with Ca: 3 g. Xa in 100 ml. liquid NH<sub>3</sub> was treated with 1.15 g. Ca, the blue soln. decolorized with AcONH<sub>4</sub>, the NH<sub>4</sub> evap., the residue treated with a satd. soln. of 3 g. (CO<sub>2</sub>H)<sub>2</sub>, the ppt. filtered off, and the filtrate passed over 25 g. Amberlite IRC-50 (in NH<sub>3</sub> cycle), the column washed with H<sub>2</sub>O, the eluate (200 ml.) evapd. to 20 ml. in vacuo at 40°, and the product (200 ml.) with EtOH gave 0.85 g. (58%) L-glutamine. L-p-

MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>NHCH<sub>2</sub>(CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CONH<sub>2</sub> (1.35 g.) reduced in 75 ml. NH<sub>3</sub> with 0.45 g. Na gave 0.63 g. (25%)  $\gamma$ -anilide of L-glutamic acid, m. 220°, (from aq. EtOH), R<sub>f</sub> in PhOH-H<sub>2</sub>O-NH<sub>3</sub> 0.89. (p-Toluenesulfonyl-a-L-glutamyl)glycine (3.58 g.) reduced with 1.4 g. Na in 120 ml. NH<sub>3</sub> and worked up with the ion exchanger yielded 1.49 g. (68.5%) N-(a-L-glutamyl)glycine, m. 175-6°, [α]<sub>D</sub><sup>25</sup> 80°. Reducing 3.58 g. of free acid from II with 1.4 g. Na in 120 ml. NH<sub>3</sub>, evapd. the demineralized soln. of the dipeptide to 20 ml., and adding 100 ml. hot EtOH gave 1.85 g. (84%) mono-NH<sub>3</sub> salt of the dipeptide which, treated with H<sub>2</sub>O, H<sub>2</sub>N-C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>OH, AcOEt and excess EtOH, yielded 95% N-( $\gamma$ -L-glutamyl)glycine, [α]<sub>D</sub><sup>25</sup> 9.4°. Reduction of 2.32 g. IX in 100 ml. NH<sub>3</sub> with 0.75 g. Na yielded 1.12 g. (70%) N-(a-L-glutamyl)-L-tyrosine, m. 186°, [α]<sub>D</sub><sup>25</sup> 31.2°. The hydrate of V (2.41 g.) reduced with 1 g. Na in 100 ml. NH<sub>3</sub> gave 0.99 g. (70.5%) dihydrate of the mono-NH<sub>3</sub> salt of N-( $\gamma$ -L-glutamyl)-L-tyrosine, m. 103-4°, also obtained in 35% yield by the reduction with Ca. II (1.48 g.) and 0.6 g. Na in 100 ml. NH<sub>3</sub> gave, by pptn. with EtOH, 0.61 g. (73%) N-(D,L-glutamyl)-glycinamide, m. 194-5°, R<sub>f</sub> (PhOH-H<sub>2</sub>O-NH<sub>3</sub>) 0.61. X (3.67 g.) reduced with 1.5 g. Na in 100 ml. NH<sub>3</sub> gave, by demineralization and pptn. with EtOH, 1.25 g. (61%) N-L-glutaminylglycine. XII. New resolution of DL-glutamic acid; synthesis of D-glutamine and ( $\gamma$ -D-glutamyl)glycine. Josef Rudinger and Hana Czurbova. Chem. Listy 48, 234-9; Collection Czechoslov. Chem. Commun. 19, 388-91 (1954).—The prepn. of p-toluenesulfonyl-DL-pyrrolidin-5-one-2-carboxylic acid (I) and its derivs. is described. Brucine was used for the resolution of I. From I, D-glutamic acid (II), D-glutamine (III), and N-( $\gamma$ -D-glutamyl)glycine (IV) were prep'd. DL-Glutamic acid (95 g.) in 317 ml. 4N NaOH treated with 122 g. p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>Cl in 320 ml. Me<sub>2</sub>CO

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and 164 ml. 4*N* NaOH gave, after filtration, removal of the *Me<sub>2</sub>CO*, and acidification with HCl, 130 g. (80%) *N*-*p*-toluenesulfonyl-DL-glutamic acid (V), m. 171-2° (from H<sub>2</sub>O); amide, m. 171°;  $\gamma$ -anilide, m. 196-7°;  $\alpha,\gamma$ -diamide, m. 213-19°;  $\gamma$ -piperidide, m. 104-5°;  $\gamma$ -hydrazide, m. 178-9°. V (17 g.) refluxed 10 min. with 50 ml. Ac<sub>2</sub>O, the excess Ac<sub>2</sub>O removed *in vacuo*, the residue dild. with 30 ml. *Me<sub>2</sub>CO*, the boiling soln. treated successively with 70 ml. H<sub>2</sub>O, and the *Me<sub>2</sub>CO* distld. off gave, after cooling to 0°, 13 g. (77.5%) I; hydrate, m. 120-8°. Anhyd. I, m. 103-70°; C<sub>6</sub>H<sub>5</sub>N salt, I, m. 163-5°; Me ester, m. 129-30°; chloride, m. 117°; anilide, m. 222-3°. I.H<sub>2</sub>O (16.07 g.) in 300 ml. hot 96% EtOH added to 19.5 g. brucine in 300 ml. 96% EtOH, and the soln. cooled and inoculated gave, after 8 hrs., 13.7 g. (78%) of the brucine salt of I, m. 222-3° (crystl. from 750-850 ml. 96% EtOH); treating 12.65 g. of the salt in 200 ml. H<sub>2</sub>O and 50 ml. AcOEt with 10 ml. concd. HCl, extg. the aq. layer with 50 and 25-ml. portions of AcOEt, washing the exts. with dil. HCl and H<sub>2</sub>O, drying, evapg. the solvent yielded 4.23 g. (82%) 1-*p*-toluenesulfonyl-DL-pyrrolidin-5-one-2-carboxylic acid (Va), m. 128-30° (from C<sub>6</sub>H<sub>6</sub>). From the mother liquors was isolated 0.55 g. (10.5%) Va hydrate, m. 08-9°. Va (5 g.) in 27 ml. 2*N* NaOH acidified with HCl after 10 min. yielded 92% *N*-*p*-toluenesulfonyl-DL-glutamic acid (Vb), m. 142-5°. Reduction of 3.01 g. Vb in 100 ml. NH<sub>3</sub> with 1.3 g. Na gave, by a procedure given in the previous paper, 1.05 g. (72%) II, [α]<sub>D</sub><sup>25</sup> -32.4°. Reducing 2.83 g. Va 15 min. with 20 ml. concd. NH<sub>3</sub> gave 2.62 g. (87%) *N*-*p*-toluenesulfonyl-DL-glutamine (VI), m. 166°. Reduction of V with Na in NH<sub>3</sub> gave 71% III, m. 180-8° (from H<sub>2</sub>O and EtOH), [α]<sub>D</sub><sup>25</sup> -7.1°. Refluxing 2.83 g. Va with 1.5 g. H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>Et.HCl, 2.5 ml. EtNC<sub>6</sub>H<sub>5</sub>, and 15 ml. MeCN 4 hrs., evapg. the solvent *in vacuo*, dissolving the residue in water, acidifying, filtering off the ppt., washing it with H<sub>2</sub>O, and purifying by repprt. from the soln. in NaHCO<sub>3</sub> yielded 3.1 g. (80%) of the Et ester of *N*-*(p*-toluenesulfonyl- $\gamma$ -DL-glutamyl)glycine (VII), m. 180-90° (from 10% Et(OH)).

Hydrolysis of VII with NaOH gave 87% free acid (VIIa), m. 186-7°. Reduction of 2.26 g. VIIa with 0.9 g. Na in 75 ml. NH<sub>3</sub> gave 1.2 g. (87%) NH<sub>3</sub> salt of IV, which yielded 78% free IV, [α]<sub>D</sub><sup>25</sup> 8.1°. The following derivs. were prep'd. as described in the preceding papers: *N*-*(p*-toluenesulfonyl- $\gamma$ -DL-glutamyl)glycine, m. 170° (Et ester, m. 170°); *N*-*(p*-toluenesulfonyl-DL-pyrrolidin-5-one-2-carboxyl)glycine, m. 192-3° (Et ester, m. 128-7°); and *N*-*(p*-toluenesulfonyl-DL-glycaminyl)glycine, m. 180-2°.

The reactive behavior of the so-called "diacopyrroles," Tommaso Ajello and Salvatore Giambrone (Univ. Palermo, Italy). *Ricerca sci.* 24, 49-54 (1954).—The chloride of *J*-diac-1,2,4,5-tetraphenylpyrrole is prepared and studied. It resembles diazonium salts in stability, oxidative power, and loss of diazonium group. Unlike diazonium salts it does not have coupling activity and loses a hydacid with alkali hydroxides and NH<sub>3</sub>.

Mario V. Conti

Decarboxylation and formylation of certain pyrrole derivatives. Edith Ju-Hwa Chu and T. C. Chu (Immaculate Heart Coll., Los Angeles, Calif.). *J. Org. Chem.* 19, 266-9 (1954).—H<sub>5</sub>NCH<sub>2</sub>CH<sub>2</sub>OH (I) is used as decarboxylation agent for substituted carboxypyroles. Adding 28.5 g. AcCH<sub>2</sub>CO<sub>2</sub>Et to Et  $\alpha$ -nitroso  $\alpha$ -benzoylacetate (from 38.5 g. BzCH<sub>2</sub>CO<sub>2</sub>Et), then adding gradually 28.5 g. Zn dust, with gentle boiling, refluxing the mixt. 2 hrs., and pouring it into H<sub>2</sub>O give 63% 2-methyl-4-phenyl-3,5-dicarbethoxypyrole, colorless prisms, m. 124-5°. 2,4-Dimethyl-3-carboxy-5-carbethoxypyrole (II), 85%, fine prisms, m. 272° (decompn.), and 2-methyl-4-phenyl-3-carboxy-5-carbethoxypyrole (III), 78%, platelets, m. 220° (decompn.) are prep'd. by partial sapon. of the corresponding diesters. Refluxing 21.1 g. II and 12.2 g. I 1 hr. and pouring the mixt. into H<sub>2</sub>O give 90% 2,4-dimethyl-5-carbethoxypyrole (IV), prisms, m. 124.5-5°; similarly, III gives 84% 2-methyl-4-phenyl-5-carbethoxypyrole, needles, m. 134.5-6°. Gradually adding 15.4 g. POCl<sub>3</sub> to 13.4 g. IV and 7.3 g. HCONMe<sub>2</sub>, refluxing

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the mixt. 2 hrs., pouring it into  $H_2O$ , and neutralizing it with NaOAc give 05% 2,4-dimethyl-3-formyl-5-carbethoxypyrrrole, needles, m. 145-6 $^{\circ}$  (oxime, m. 190-200 $^{\circ}$ ; semicarbazone, decomp. at about 276 $^{\circ}$ ). 2-Methyl-4-phenyl-3-formyl-5-carbethoxypyrrrole, prep'd. similarly in 97% yield, prisms, m. 144.5-5 $^{\circ}$  (oxime, 98%, plates, m. 210-11 $^{\circ}$ ; semicarbazone, needles, decomp. at about 280 $^{\circ}$  with sublimation).

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Amino acids and peptides. Part 11. A new synthesis of peptides of glutamic acid [in English with summary in Russian]. Sbor.Chekh.khim. rab. 19 no.2:375-385 Ap '54. (MLRA 7:6)

1. Department of Organic Synthesis, Institute of Organic Chemistry, Czechoslovak Academy of Science, Prague.  
(Glutamic acid) (Peptides)

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Amino acids and peptides. Part 12. A new resolution of dl-glutamic acid; synthesis of d-glutamine and  $\gamma$ -d-glutamylglycine [in English with summary in Russian]. Sbor.Chekh.khim.rab. 19 no.2:386-392 Ap '54. (MLRA 7:6)

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Synthesis of Peptides of Glutamic Acid", P. 235, (CHEMICKÉ LISTY,  
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SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 3,  
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From L-glutamic acid. Zdenek Pravda and Josef Rindinger C  
 (Czech. akad. věd, Prague). Collection Czechoslov. Chem. Commun. 20, 1-8 (1955);  
 English; cf. C. I., 49, 3126. — Treating  $\text{L-TsN}(\text{CH}_3)\text{COCl}$  (I) ( $\text{Ts} = p\text{-MeC}_6\text{H}_4\text{SO}_2$ ) with  $\text{NH}_2$ , gave  
 $\text{L-TsN}(\text{CH}_3)_2\text{CHCONH}_2$  (II).  $\text{L-TsN}(\text{CH}_3)_2\text{C}$   
 $\text{HCONHPH}$  (IIIa) was prep'd. from II. Both II and IIIa were  
 reduced by  $\text{LiAlH}_4$  or, in better yields, with  $\text{LiBH}_4$  to  
 $\text{L-TsNHCH}(\text{CONH}_2)\text{CH}_3$ , resp. Heating IIIa with  $\text{K}_2\text{CO}_3$  gave  $\text{L-TsN}(\text{CH}_3)_2\text{CHCONH}_2$  (IVa). Similar  
 treatment of III failed to give  $\text{L-TsN}(\text{CH}_3)_2\text{CHCONH}_2$ .  
 (IV). To prep. IV, III had to be transformed to  $\text{L-TsN}(\text{CH}_3)_2\text{CHCONH}_2\text{Br}$  (V) which, heated with  $\text{K}_2\text{CO}_3$ , gave  
 also prep'd. from  $\text{TsCl}$  and  $\text{NH}_2$  in  $\text{NaOH}$  gave  $\text{L-proline}$  (VI). Reduction of VI,  
 IVa with  $\text{NaBH}_4$  in  $\text{NH}_3$  gave  $\text{L-TsN}(\text{CH}_3)_2\text{CHCO}_2\text{H}$  (VII). M.p.s. are corrected. The above reactions repre-  
 sent the 1st chem. transformation. L-proline. M.p.s. are corrected. I, prep'd. according to